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Fast-Burning Rate/High Slope Propellant Technology Program Final Report [U]

by

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and

A. Katzakian

Aerojet Solid Propulsion Company (ASPC)

for the

Propulsion Development Departr..ent

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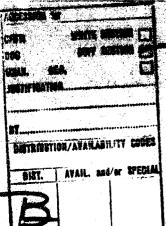
Naval Weapons Center

CHINA LAKE, CALIFORNIA & SEPTEMBER 1971



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ABSTRACT

(U) Two candidate propellant formulations, ANB-3394 and ANB-3395-1, were developed that satisfied all the technical goals. Additionally, these propellants were successfully test fired in small scale motors verifying propellant ballistic properties, excellent liner propellant bonds, and propellant processability adequate for good grains.

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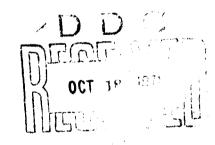
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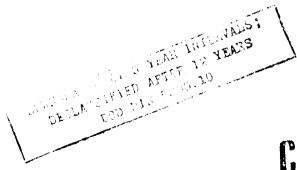
This final report describes work conducted by Aerojet Solid Propulsion Company, Sacramento, California for the Naval Weapons Center, China Lake, California during the period 1 May 1970 through 22 April 1971 under Navy contract NOO123-70-C-1457. This work was supported by the Naval Air Systems Command under AirTask WF 19.332.302.

 $\,$ M. F. Pickett of NWC was technical coordinator for this program and has reviewed this report for technical accuracy.

Released by RAY MILLER, Head Propulsion Systems Division 1 September 1971 Under authority of G. W. LEONARD, Head Propulsion Development Department

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INTRODUCTION

- (U) The objective of this 11-month program was to advance the state-of-the-art with regard to formulation of practical fast-burning and high-pressure-exponent propellants by expanding available technology.
- (U) The specific goals were the formulation of two propellants, hereinafter referred to as "A" and "B", with respective burning rates of 3.5 and 7.0 in/sec. at 2000 psia and a pressure exponent of about 0.70. Both propellants were to be formulated to deliver a specific impulse (1000) of at least 240 lbf-sec/lbm with a density of 0.063-0.065 lbs/in.3. The study also included the development of adequate mechanical properties to withstand the temperature range of -40 to 160°F. Other considerations were adequate processing, potlife (4 hrs. @ 135°F), thermal and aging stability and safety characteristics. Only composite propellants were to be considered, with porous AP (PAP) and non-volatile ferrocene derivatives limited to the high burning rate propellant "B".
- (U) As a final part of the program ten grains measuring 2 in. dia. \times 6.25 in. length and four grains measuring 4.8 in. dia. \times 15 in. length of each candidate formulation as well as representative samples of these propellants were forwarded to NWC, China Lake, for further testing.

SUMMARY

- (U) Two candidate propellant formulations, ANB-3394 and ANB-3395-1, were developed on this program that satisfied all the technical goals. Additionally, those propellants have been successfully test fired in small scale motors verifying propellant ballistic properties, excellent liner propellant bonds, and propellant processability adequate for good grains. All required grains have been delivered to NWC, China Lake.
- (U) Two propellant formulations, ANB-3361 and ANB-3364, from the previous high burning rate/high slope program on Contract No. N00123-69-C-0401 were chosen as baseline formulations. Both baseline formulations were processed at the beginning of this program and were found to be deficient in processing, mechanical, and ballistic properties.

- (U) Improvement in processing properties (viscosity, potlife) was considered to be the key to a successful program because it is a necessity for the processing of large batches for delivery grains and that excess processability may be traded for a higher burning rate. The problem of short potlife was solved by using a modified form of the prepolymer R-45M. This modification consisted in blocking excess hydroxyl groups with a monoisocyanate which resulted in a three fold increase in potlife for ANB-3394. However, ANB-3395-1, the Catocene containing formulation, did not show improvement in potlife using modified R-45M until acetylacetone (HAA) was also added to the formulation. The Fe⁺⁺⁺ contamination in the Catocene was catalyzing the isocyanate-hydroxyl reaction and the HAA served to suppress this catalytic effect through chelation.
- (U) Improvements in propellant mixing and mechanical properties were achieved with the incorporation of the functional wetting agent DEO and the bonding agent TEA. These ingredients also made the incorporation of finer exidizer and burning rate catalysts easier, while maintaining or improving mechanical properties.
- (U) Significant increases in burning rate were achieved by (1) increasing the propellant mix cycle, (2) replacing the 10μ NH₄ClO₄ with 5μ NH₄ClO₄ and, (3) in the case of ANB-3394, replacing the standard Fe₂O₃ with a finely ground, crystalline Fe₂O₃. For ANB-3395-1, the desired burning rate was achieved by increasing the Catocene to 4% from 3%, and using a more active porous ammonium perchlorate (PAP).
- (U) Hazard characteristics of the final formulations were determined in both the uncured and cured states. No unusual processing or handling characteristics were indicated. One batch of ANB-3395-1, however, having a high Shore reading (74), exhibited high friction sensitivity, but all subsequent batches with shore hardnesses of less than 60 showed much greater stability to friction. Both candidate propellants were classified as Class "B" explosives by ICC tests.
- (U) Two 175 1b batches, one each of ANB-3394 and ANB-3395-1, were processed from which $\sin z 5 \times 19$ in. phenolic sleeves were cast from each batch using an improved casting setup; all grains cast were void-free by radiographic inspection. Also, void-free gallon cartors and DPT specimens were cast from each batch to measure mechanical, ballistic and bonding properties. This demonstrated that both candidate formulations have adequate processing characteristics.
- (U) Reproducibility in mechanical properties and burning rates were good from batch to batch for each candidate formulation. After one month aging at 135°F each formulation showed increases in tensile strength with small decreases in elongation; burning rates as measured in solid strands appeared unchanged and liner/propellant bonds remained excellent.

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(U) Several small grains were test fired to substantiate solid strand burning rates, propellant/liner bond and grain integrity. Initial firings resulted in erratic pressure-time traces due to aluminum deposition on the nozzles. The following changes were effected to minimize such deposition: (1) Increase L* by shortening grain to 3 in., (2) cast a 0.25 in. layer of non-aluminized propellant on the main grain to serve as a nozzle warmer, (3) use a contoured boron nitride nozzle, and, (4) change from H-95 to H-60 aluminum. After these changes had been made, good motor firings were obtained which verified the corresponding solid strand burning rates.

TECHNICAL DISCUSSION

- (U) The candidate formulations from the previous NWC contract N00123-69-C-0401¹, ANB-3361 and ANB-3364 (Table 1), were selected as baseline formulations, respectively, for propellant "A" (ANB-3394) and propellant "B" (ANB-3395-1). The processing and mechanical properties of the baseline formulations were marginal, and since the target burning rates for propellants "A" and "B" for this program were about 20% higher than the rates for the baseline formulations (Figure 1) we decided to make improvements in processing and mechanical properties first, and then trade them off as needed to make the necessary ballistic improvements. Also, the work centered largely on meeting propellant "A" goals since such progress could be readily used to achieve the corresponding goals for propellant "B".
- (U) Since poor propellant/liner bonding was the suspected cause of motor failures in the previous program, early demonstration of good liner bonds and motor firings was planned and demonstrated.

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¹⁽U) Aerojet General Corporation. Fast-Burning Rate/High Slope Propellant Technology Program Final Report 15 March 1969--15 January 1971. Sacramento, California.

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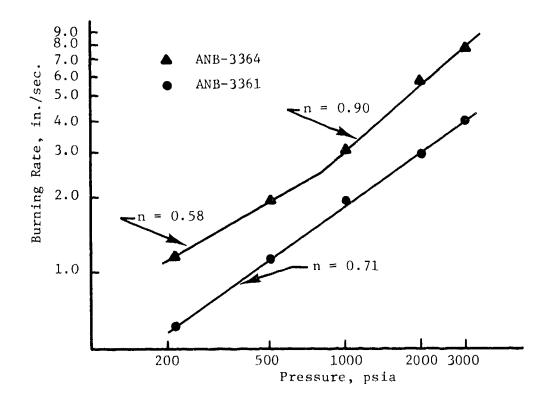
(C) TABLE 1. Composition of Candidate Formulations Developed for NWC Contract NOO123-69-C-0401

Composition, %

Ingredients	ANB-3361	ANB-3364
NH ₄ C10 ₄ (0.5μ) ^a	50.00	35.00
NH ₄ C10 ₄ (7-10μ)	19.00	25.00
Porous NH ₄ ClO ₄ (180μ)	•••	10.00
Aluminum (H-60)	15.00	15.00
Fe ₂ 0 ₃	0.50	
BRA-101	0.50	
HYCAT-6		3.00
Plastinox #711	0.30	0.10
Agerite White	0.20	0.20
Sulfur		0.30
Oronite 6	4.50	
R-45M	9.28	1.00
HT-Telagen		9.76
TEA	0.04	
C-1		0.05
IPDI	0.68	0.59
	100.00	100.00

^aBDB coated, 0.5%.

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(U) FIG. 1. Plot of Solid Strand Burning Rates vs. Pressure for Candidate Batches from Previous NWC Contract NOO123-69-C-0401.

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PROCESSING AND MECHAN CAL PROPERTY STUDIES

(U) The problems of poor processing and mechanical properties were solved by a combination of approaches involving the use of a functional wetting agent, that also serves as a bonding agent, in combination with a functionally modified prepolymer and second bonding agent. For the Catocene containing propellant the inclusion of a chelating agent greatly increased the potlife. Various other approaches to improving processing and mechanical properties were explored, some moderately successful and others unsuccessful. All the approaches are presented and discussed in the following sections.

Evaluation of Wetting and Bonding Agents

- The addition of diethanololeamide (DEO) into the ANB-3361 formulation resulted in greater ease of mixing, a smoother, less pasty appearance to the propellant, noticeably better castability and mechanical properties, but little or no improvement in potlife. The DEO approach was evaluated further in conjunction with TEA. The results of these studies are summarized in Table 2. Inspection of the table reveals some interesting trends. For example, when TEA was eliminated from the formulation a loss in elongation was noted although mixing and casting properties were unchanged. When TEA and DEO were both removed mixing, casting and mechanical properties were all impaired. Having established the need for TEA and DEO, we determined those levels of each that yielded optimum processing and mechanical properties. Referring to Table 2 again, a significant upward trend in mechanical properties was observed with the stepwise increase of DEP at the expense of R-45, holding TEA and IPDI constant. Both tensile strength and elongation increased implying that the DEO was also functioning as a bonding agent. Unfortunately, at the high DEO level the potlife was extremely short resulting in poor castability and a porous sample that prevented the measurement of mechanical properties. The data suggest, however, that an optimum level of DEO is about 0.2% of the formulation corresponding to an equivalence level between 10 and 15. The optimum TEA level is indicated at .02% of the formulation which corresponds to about 5 equivalents. A lower IPDI level is also indicated since a lower modulus and higher elongation is desired.
- (U) The addition of TEA after IPDI addition in the mix cycle resulted in a slight increase in elongation with a drop in tensile strength and modulus (Table 2). This technique has shown increases in both tensile strength and elongation in other propellants, but appears here to have simply reduced the effective IPDI level by reaction with ammonia released from TEA-NH $_4$ ClO $_4$ interaction, since similar results would be expected.

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TABLE 2. Effects of Wetting and Bonding Agents on Processing and Mechanical Properties

(C)

		Binder Variations	riations			Mechanical Properties @ 77°F	Propertie	s @ 77°F	
Batch No. AK-	д-45М (Equiv.)	TEA (Equiv.)	DEO (Equiv.)	IPDI (Equiv.)	Mixability	σ m, psi	ε <mark>m,</mark> %	E _o , psi	Shore A Hardness
7365-18	06	10	:	80	Poor	165.4	16.7	1001	72
7365-20	06	5	5	80	Fair	154.6	18.9	864	65
7365-22	06	:	10	80	Fair	144.5	15.6	976	70
7365-41	85	10	5	80	Fair	177.5	17.8	1055	:
7365-43	80	10	10	80	Good	194.4	21.7	1071	:
7365-45	70	10	20	80	Good	213.5	20.6	1198	:
7365-63	50	10	70	80	Fair	:	:	:	:
7365-49 ^a	80	10	10	80	Good	156.2	22.2	764	:
7365-53	80	10	10	75	Good	173.0	22.8	853	:
7365-55	80	10	10	70	Good	128.1	23.4	627	:
7591-36b,c	100	•	•	70	Poor	81.6	18.5	7.4	52
7591-38b,c	95	5	:	70	Fair	92.2	19.2	507	99
7591-40b,c	85	5	10	70	Good	118.0	19.4	674	99

Any benefits in castability that would have resulted from the above variations were masked by very short potlife. All batches contain (50%) 0.5ν UFAP, (19%) MA-AP, (15%) Al-H60, (0.5%) E₂03, (0.5%) Silon S, (5.0%) Oronite 6 and aging stabilizers unless noted otherwise. NOTE:

 $^{\mathrm{a}}.\mathrm{FA}$ added last in mix cycle.

 $^{
m b}_{
m Burning}$ rates at 2000 psia and pressure exponents (n) for AK-7591-36, -38, -40 respectively are 3.52 (0.76), 3.40 (0.74) and 3.48 (0.78).

 $^{ extsf{C}}$ Contains 5 $_{\odot}$ UFAP in place of MA-AP and Al-H95 in place of Al-H60.

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(U) Although this formulation has responded favorably to bonding agents, the effects were not as large as was expected. We suspected, therefore, that the BDB coating on UFAP may either be interfering with the bonding agent, or function as a bonding agent itself. This hypothesis was tested by comparing 0.9μ UFAP coated with 0.5% Victoria Blue R (a rosanilin dye) against 0.9μ UFAP coated with 0.5% BDB. The dye coated UFAP mixed and cast with more difficulty and gave a lower burning rate at 2000 psia than the BDB coated UFAP, probably due to differences in deagglomeration rates.

	0.9µ UFAP (0.5% BDB)	0.9μ UFAP (0.5% Dye)
r _b , in/sec @ 2000 psia	2,92	2.73

- (U) Although mechanical properties were not measured, it was clearly evident that the BDB coated UFAP produced a propellant with superior mechanical properties.
- (U) An unsuccessful attempt to further enhance the beneficial properties of DEO was made by adding a non-functional co-wetting agent such as lecithin to the formulation. This wetting agent adversely affected both the mixing and casting characteristics of the propellant. No further work with co-wetting agents was conducted.

Evaluation of Alternate Prepolymer and Curing Systems

- (U) Because of the short potlife exhibited by the R-45M/IPDI binder, alternative prepolymer and curing agents were evaluated in the "A" formulation. However, no significant improvements in processing, potlife or mechanical properties were produced by these systems.
- (U) <u>Carboxyl-Terminated Polybutadiene/Epoxide System</u>. The use of HC-434 carboxyl-terminated polybutadiene with epoxide curing agents such as ERL-4221 or ERL-4205 did not improve the potlife of the "A" formulation.
- (U) R-45M/Imine System. Utilization of the diimine, BISA, as a curing agent for R-45M in the "A" formulation afforded negligible potlife and resulted in a hard, brittle propellant mass.
- (U) R-45M/Epoxide Systems. The application of ERL-4206 epoxide as a curing agent for R-45M provided a 4 hour potlife at 135°F for the "A" formulation. However, a satisfactory elastomeric cure was not achieved. Replacement of ERL-4206 with ERL-4221 in the "A" formulation resulted in a negligible potlife and a brittle propellant.
- (U) R-45M/Epoxide/IPDI Systems. The use of various epoxides in combination with IPDI were evaluated with the goals of extending potlife and providing propellant with adequate mechanical properties. With the IPDI held at 35 eq. the following diepoxides were formulated at the 65 eq. level in the "A" formulation:

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Epoxide	Batch No.
None	AK-7591-24C
Resorcinoldiglycidyl ether (CIBA	AK-7591-24D
ERE 1359)	
Vinylcyclohexene dioxide (Union	AK-7591-24E
Carbide ERL-4206)	

- (U) The data from these experiments are summarized in Table 3 and show that no significant improvements in potlife or mechanical properties were gained from the epoxide/IPDI curing systems.
- (U) <u>Cure Retarders</u>. Experience at ASPC in other propellants has shown that trioctylphosphine oxide (TOPO), phenol and trioctylphosphate (TOF) can be used to extend the potlife of polyurethane propellants. The use of TOPO (0.3%), phenol (0.2%), or TOF (4.5%, as a replacement for the Oronite 6 plasticizer), however, did not increase the potlife of the "A" formulation.
- (U) <u>Blocked Isocyanate</u>. Blocked isocyanates have been used to delay polyurethane cure reactions. In order to obtain a blocked isocyanate, TDI was combined with the blocking agent, acetylacetone, for 48 hours prior to use in the propellant. However, no increase in the potlife of the "B" propellant was observed when the blocked TDI was used in place of IPDI.
- (U) $\underline{\text{Dimerized Isocyanate}}$. TDI dimer was obtained via the following reaction.

The dimer was formed by blending 1 mole of TDI in 500 ml toluene with 5 drops of tri-n-butylphosphine as the catalyst. The mixture was stirred overnight and the resulting solid was collected by filtration and washed well with hexane. The vacuum dried material had a m.p. of 154-156°C, identical to the literature value. The I.R. spectrum was also in accord with the expected dimerized structure, giving a strong isocyanate absorption at 2280 cm⁻¹ and a strong urea-like carbonyl absorption at 1772 cm⁻¹. The propellants made with this material gave reasonable potlifes, but cured rapidly to a hard, brittle material with poor mechanical properties. (Table 3, #AK7591-17, -24A, -24B) Since no advantage in potlife was demonstrated (Figure 2, #AK7591-24A), and since the resultant mechanical properties were poor, no further work was done with this material.

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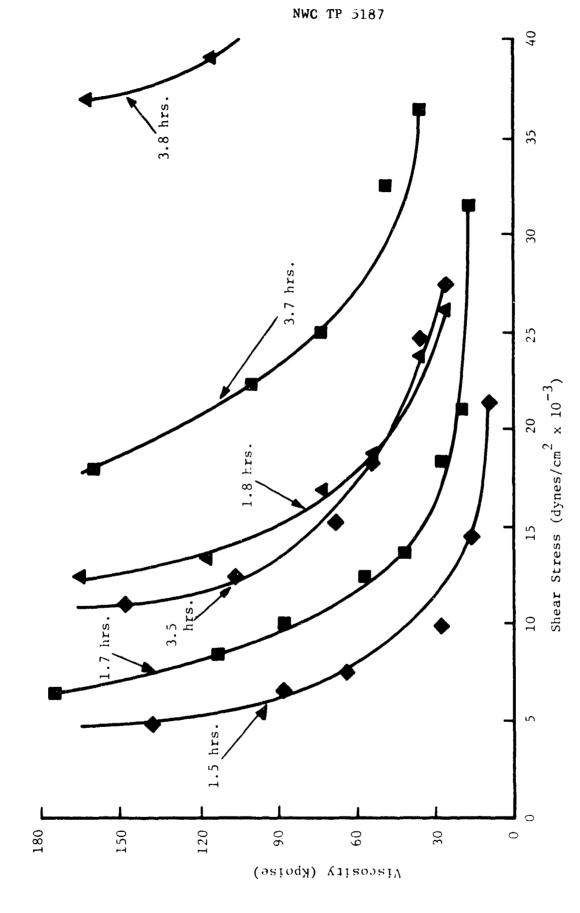
(C) TABLE 3. Effect of Dimerized TDI and Epoxide Curing Agents on Processing and Mechanical Properties

	Shore A Hardness	59	12		20	38	5		0	25		10	
	Potlife, hrs ^a	•	•		:	1.5	1.0		3.0	1.8		1.5	
,77°F	E _o , psi	516	282		_		:		:			:	
Mechanical Prop.,77°F	em, %	19.6	21.9		Poor Properties	Poor Properties	:		•			•	
Mechar	σm, psi	89.5	43.7		Poor F	Poor F	:		:			:	
op.,80°F	u	82.0	0.76		•	:	•		•			•	
Ballistic Prop.,80°F	*2000' ips	3.25	2.98		•	•	•		•			•	
	Curing Agent (Equiv.)	(70) IPDI	IPDI/4206	(32)/(62)	TDI-Dc(70)	TDI-Dc(65)	TDI-DC/4206	(32)/(62)	IPDI (35)	TPDI/RDE	(32)/(62)	IPDI/4206	(35)/(65)
	AP Coarse Fraction, μ	3	3		٣	&	8		&	œ		<u></u>	
	Batch No. AK-	7591-11 ^b	7591-13 ^b		7591-17	7591-24A	7591-24B		7591-24C	7591-24D		7591-24E	

All propellants contain 50% 0.4 UFAP, 19% 3 -8 UFAP, 15% Al-H95, 0.5% Fe₂0₃, 0.5% Silon-S, 15% R-45M/IPDI binder, except where noted, and mix cycles have been set at ²4 hours. NOTE:

 $^a\mathrm{Time}$ to 50 kpoise 120°F, 10,000 dynes/cm 2 Shear Stress $^b\mathrm{Contained}$ IDP in place of Oronite 6.

^cDimerized TDI.



(U) FIG. 2. Viscosity vs. Shear Stress at 120°F and Two Time Intervals. The Time Noted on Curves Denotes Time After IPDI Addition. Curves include (1) ■ Dimer TDI (AK7591-24A), (2) ▲ IPDI 35 eq./4206 65 eq. (AK7591-24E) and (3) ◆ IPDI 35 eq. (AK7591-24C).

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Effect of Plasticizer Type and Level

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- (U) Because of the much lower viscosity of IDP relative to Oronite 6, it was of interest to determine whether or not IDP would improve processing and mechanical properties. Consequently, a batch of propellant "A" was prepared wherein all the Oronite 6 was replaced with IDP. Although this change resulted in a more processable propellant, no improvement was observed in mechanical properties and the burning rate of the cured propellant was adversely affected (Table 4, #AK7591-11). The decrease in tensile strength was not explainable and may not be a result of using IDP. The depressing effect on burning rate was attributed to the ester group in IDP since such groups do tend to give lower burning rates at high pressures (1000 2000 psi). Since losses in burning rate could not be tolerated, IDP was not considered further.
- (U) Increasing the Oronite 6 plasticizer from 4.8 to 8.6% did not significantly increase the potlife of the "A" formulation (Table 4, #AK7591-52). Moreover, the burning rate and mechanical properties were adversely affected by the additional plasticizer. Efforts in this direction were discontinued.
- (U) Due to the processing improvements that have been made in propellant "A" (TEA-DEO system), lower plasticizer levels were evaluated to see if mechanical properties could be improved while maintaining adequate processability (Table 4, AK7591-79, -81, -83, -85). Unfortunately, only increases in tensile strength were achieved, elongation remaining constant. Moreover, the propellant viscosity increased significantly at the lower plasticizer levels as shown in Figure 3. A comparison of viscosity buildup vs. time under a shear stress of 10,000 dynes/cm² at 120°F is presented in Figure 3 for three plasticizer levels. The potlife, measured as time to 50,000 poise, falls off rapidly as plasticizer decreases. Primarily, this is a result of higher initial viscosities, since the rates of viscosity buildup are similar. Going to higher cast temperatures lowers these initial viscosities, but due to rapid buildup rates, the useful potlife is shortened (Figure 4).

Effect of Aluminum Particle Size, Ballistic Solids And IPDI Level

(U) An enhancement of the mixing operation was realized when the coarser H-95 aluminum was used to replace the H-60 aluminum. The castability was also somewhat better, but the short potlife tended to obscure improvements in this area. No significantly adverse effects were observed on the burning rate.

Pressure	r _b , in/	sec
riessure	A1-H60	A1-H95
500 psia 2,000 psia	1.04 2.92	1.03 2.86

NOTE: Slope of both propellants 0.74.

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(C) TABLE 4. Effect of Plasticizer Type and Level on Processing, Ballistic and Mechanical Properties

	Shore "A" Hardness	59 78 62 18 62 62 45 53	
	Potlife, ^a Hrs	1.5 3.0 3.0 1.3 1.5 1.5	
p.,77°F	E _o , psi	516 1357 635 242 251 770 540 605 735	
Mechanical Prop.,77°F	, ш %	19.6 11.0 18.3 30.9 29.1 15.4 19.1	
Mechan	σm, psi	89.5 149 107 50.2 52.1 109 86.5 93.8	
op.,86°F	и	0.78 0.78 0.78 0.75 0.75 0.77	
Ballistic Prop.,80°F	^r 2000 in/sec	3.25 3.32 3.40 3.40 3.40 3.45 3.45	
iables	Oronite-6 (%)	4.80b 8.00 4.80 4.50 4.50 4.50 3.50	
Binder Variables	DEO (Eqv)	20 10 10 10 10 14 15 14	
Bir	R-45M (Eqv)	75 85 85 85 85 80 78 80 81	
	Batch No. AK 7591-	11 55 56 62 76 79 81 83 83 85	

All batches contain (50%) 0.55 UFAP, (19%) 5 UFAP, 15% Al H-95, and 15% binder with TEA and IPDI held constant at (5 eq) and (70 eq), respectively, unless otherwise noted. cycles are 4 hours. NOTE:

 $^{\rm a}_{\rm Tim.}$, to 50 kpoise at 120°F and 10,000 dynes/cm $^{\rm 2}$ shear stress.

^bUsed IDP in place of Oronite 6.

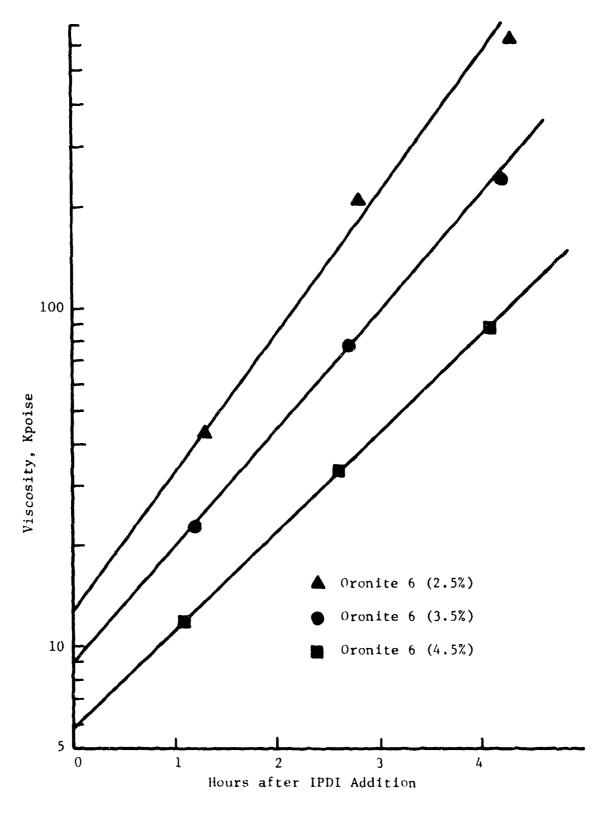
Cused new can of same lot of R-45% as used in all other batches.

 $^{\rm d}_{\rm Modified}$ R-45M calculated as unmodified R-45M and designated R-45M-50

 $^{\rm e}$ _{Same} as (d) but used R-45M-25, 65 equiv. IPDI, ground crystalline Fe $_2$ 0 $_3$, (40%) 0.55 $_{\circ}$ UFAP and (29%) 5° UFAP.

 $f_{contains}$ (45%) 0.55% UFAP, (24%) 5% UFAP, modified R-45M, .02% TEA and 100 equiv. IPDI.

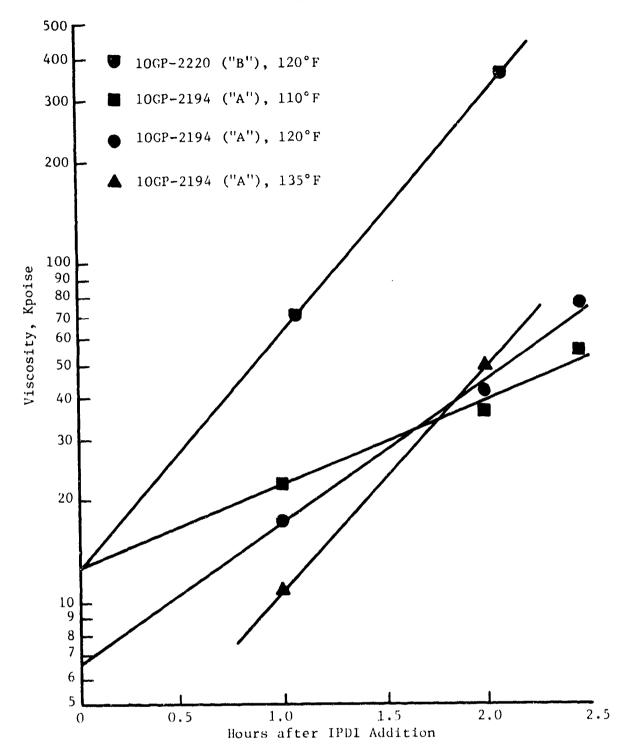
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(U) FIG. 3. Effect of Oronite 6 Level on Propellant "A" Viscosity vs. Time at 10,000 dynes/cm 2 Shear Stress and 120°F.

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(U) FIG. 4. Viscosity Buildup of Candidates "A" (110° F, 120° F, and 135° F) and "B" (120° F), at 10,000 dynes/cm² Shear Stress.

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H-95 Al was used until near the end of the program when nozzle deposition was shown to be a problem in small motor testing. The final formulations used the finer H-60 Al.

- (U) Because of the long interim mix cycle and the coarse aluminum used in these propellants, it was desirable to know whether or not the aluminum was producing any side effects as a result of abrasion. One batch was therefore prepared in which the H-95 Al was added after the interim mix cycle. The data (Table 5, #AK7591-26) indicate a slight lowering of the burning rate and a somewhat harder cure than is normally seen. The lower burning rate could be due to less efficient breakup of the UFAP particles and the harder cure may be attributed to less reaction of the fresh aluminum surface with the hydroxyl groups thereby allowing more complete reaction with the isocyanate curing agent. The effect is small enough, though, to be considered as a normal variation for this propellant.
- (U) Two batches were made in which the ballistic solids were lowered by one and two percent, respectively (Table 5, #AK5691-42 and -28). Significant improvements in processing and mechanical properties were seen, especially for the 83% solids formulation. The sacrifice in burning rate, however, was too great to give serious consideration to this approach to improving processing and mechanical properties.
- (U) Observation that residual R-45M on the outside of the container eventually formed a polymer led to the speculation that oxidative crosslinking may be slowly causing a viscosity increase in the R-45M being used. An unopened can of the same lot of R-45M was evaluated to see if any differences in processing were apparent (Table 5, #AK7591-56). Although the end of mix viscosity data show no significant differences, due to rapid cure, there was a noticeable improvement in the mix viscosity indicating that the exposed container of R-45M had undergone a small amount of oxidative crosslinking.
- (U) Since previous work had shown that 70 equivalencs of IPDI still produced too hard a propellant (Table 5, #AK7365-71) and current work (Table 5, #AK7591-24C) showed a significant increase in potlife by lowering IPDI, a series of three batches were prepared (Table 5, #AK7591-30, -32, -34) varying the IPDI from 45-65 equivalents. Rotovisco data (Figure 5) were taken on these batches to determine the viscosity buildup at each IPDI level. The data indicated that an IPDI level of 55-60 equivalents would be desirable from the standpoint of processing and mechanical properties.

Improved Potlife With Modified R-45M and Acetylacetone (NAA)

(U) No significant increase in the potlife of propellant "A" was realized until modified R-45M was used in place of R-45M. This modification was accomplished by blocking a portion of the hydroxyl groups on R-45M with a mono isocyanate, hence, the designation R-45M-25 means 25% of the hydroxyl groups are blocked; R-45M-35 means 35% are blocked, etc. Using R-45M-25 resulted in an increase in potlife (Table 6, #AK7591-76, -70) two and one half times that obtained with

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(C) TABLE 5. Effects of Varying Solids and IPDI Level on Processing, Mechanical and Ballistic Properties

	Formulation Variables	Variables	Ballistic l	Ballistic Prop.,80°F	Mechan	Mechanical Prop.,80°F	2.,80°F		
Batch No. AK-	Solids Level (%)	rpDI Level (Equiv)	$^{r}_{2000}$ in/sec	u	cm, psi	ε m, %	Eo, psi	Potlife ^a (Hrs.)	Snore "A" Hardness
7591–26 ^b	85	02	3.37	0.76	153	19.9	847	1.0	75
7591-42 ^c	84	70	3.25	0.76	129	21.8	099	2.0	89
7591-28 ^d	83	70	3.07	0.72	147	30.5	563	3.0	29
7591-56e	85	70	3.40	0.78	107	18.3	635	1.0	62
7365-71 ^f	98	70	3.05	0.76	129	18.3	760		7.1
7591-24C ⁸	85	35	:	:	:	•	:	3.0	0
7591-30	85	45	3.28	08.0	55.1	33.8	243	1.0	18
7591-32	85	55	3.39	0.79	71.2	25.7	344	0.8	33
7591-34	85	65	3.51	0.78	120	14.2	935	0.5	65

All formulations contain (50%) 0.55 μ UFAP, (19%) 5 μ UFAP, (15%) A1-H95, and (15%) R-45M/IPDI binder with TEA, DEO and R-45M held constant at 5, 20 and 75 equivalents respectively, unless otherwise noted. NOTE:

 2 Time to 50 kpoise at 120°F and 10,000 dynes/cm 2 shear stress.

 $^{
m b}$ Aluminum added one half hour prior to IPDI addition.

 $^{\mathsf{C}}_{\mathsf{Aluminum}}$ decreased two percent and 5μ UFAP increased one percent.

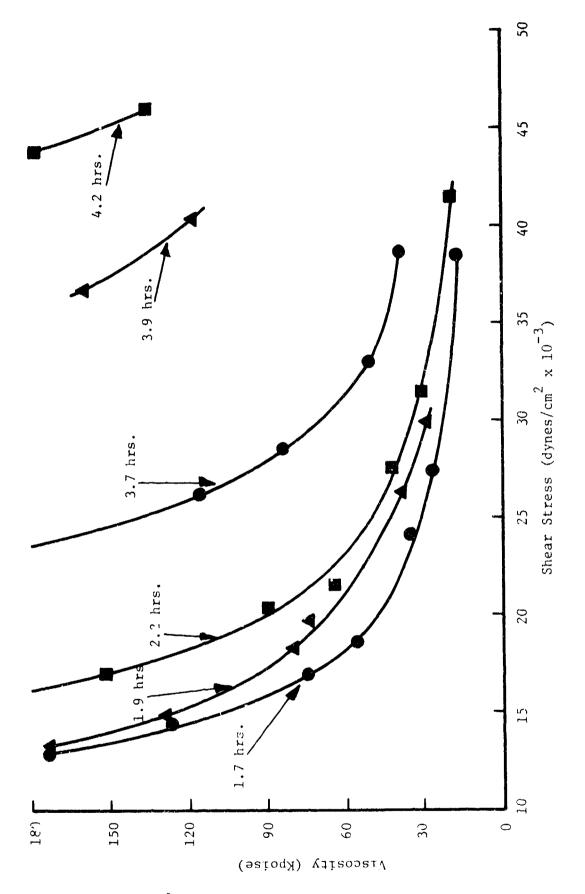
dAluminum decreased two percent.

^eUsed new container of the same lot R-45M used in other batches.

fused 20% $\mathrm{NH_4C10_4}$ (MA) in place of 5μ UFAP.

 $^{
m S}$ Used NH $_{4}^{
m C10}_{4}$ (MA) in place of 5 μ UFAP.

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The Time Noted on Curves (U) FIG. 5. Viscosity vs. Shear Stress at 120°F and Two Time Intervals. The Time Noted on Curves Denotes Time After IPDI Addition. Curves Include (1) IPDI 45 eq. (AK7591-30), (2) ■ IPDI 65 eq. (AK7591-32) and (3) ■ IPDI 65 eq. (AK7591-34)

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(C) TABLE 6. Effect of Modified R-45M and Acetylacetone (HAA) on Potlife and Nechanical Properties

Ĺ	Potlife ^a Shore "A" (Hrs) hardness	2.0 44	5.0 28	3.2 45	5.0 18	70	1.2 48	2.8 43
p.,77°]	E _o , psi	353	349	535	242	781	229	483
ical Pro	εm, %	24.1	25.7	19.2	41.8	16.7	16.8	22.5
Mechan	om, psi	9.92	78.4	86.3	50.2	115	85.6	78.7
Ballistic Prop.,80°F Mechanical Prop.,77°F	n	0.75	0.75	0.77	0.78	06.0	06.0	0.88
Ballistic	*2000 in./sec	3.38	3.39	3.40	3.38	6.78	6.70	6.65
	Formulation Type	"A"	', Y';	''A''	''A''	: Q :	g.,	ୁ, ଯ
	HTPB (Type)	R-45M	R-45M-25	R-45M-35	R-45M-50	R-45M-25	R-45M-35	R-45M-35
	Batch No. AK-	7591-70	7591-76	7591-81	7591-62	7591-89	10GP-2248	10GP-2457 ^b R-45M-35

All "A" formulations contain 69% (0.5 and 5 μ) UFAP, 15% Al, 0.5% Fe₂0₃, 0.5% Silon S and 15% HTPb binder. The "B" formulations contain 70% AP (0.5 μ , MA, and PAP), 15% Al, 4% catocene and 11% hTPB binder. NOTE:

 $^{\rm a}{\rm Time}$ to 50 kpoise at 120°F and 10,000 dynes/cm $^{\rm 2}$ shear stress.

^bContains 0.2% HAA.

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unmodified R-45M). A graphic illustration of this potlife effect is shown in Figure 6, where viscosity buildup for the two batches is measured with time at two different shear stresses.

as a reasonable compromise to achieve reasonable potlife, with adequate cure and mechanical properties. These experiments are summarized in Table 6. Although propellant "A" responded to modified R-45M with a significant potlife increase, such was not the case for propellant "B". Assuming that the catalytic effect of Fe⁺⁺⁺ contamination in the Catocene was overriding the potlife effect of the modified R-45M, acetylacetone (HAA) was added to the formulation to deactivate this catalytic species by chelation. This resulted in a potlife (Table 6, #10GP-2457) comparable to that achieved for propellant "A" with modified R-45M. The small drop in mechanical properties noted for the HAA containing batch was attributed to incomplete cure, since later aging results have yielded almost identical properties to the "B" formulation without HAA (Table 6, #10GP-2248). The conclusion was that HAA has no significant effect other than increasing potlife.

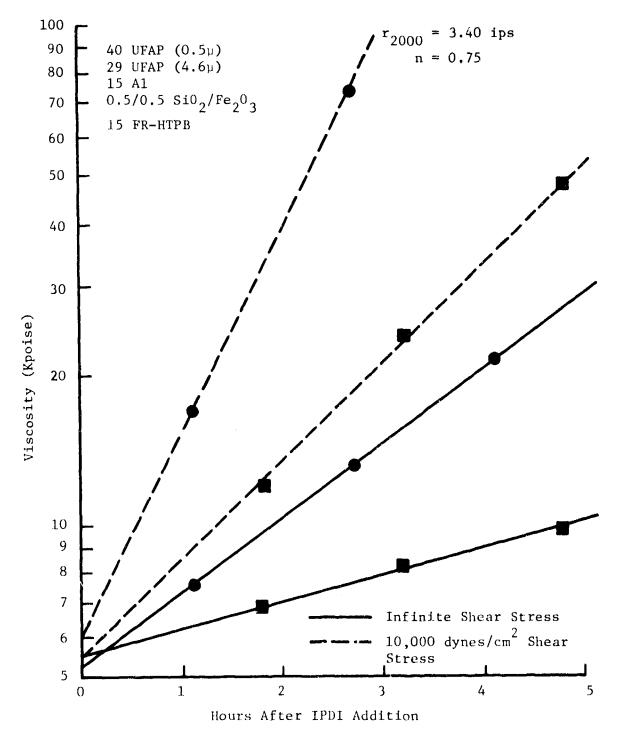
BALLISTIC STUDIES

(U) Because of the previously described processing improvements, finer oxidizer blends as well as very fine iron oxide were able to be processed into propellant "A", which in combination with a long mix cycle yielded the desired burning rate of 3.5 in./sec at 2000 psia. Likewise the goal of 7.0 in./sec for propellant "B" was very nearly realized. In this case attainment of the desired burning rate was made somewhat less difficult because porous ammonium perchlorate (PAP) and Catocene were allowed in the formulation.

Importance of Amount and Particle Size of Ammonium Perchlorate

As in the processing and mechanical properties study, we (II) felt that any progress made on propellant "A" could be applied toward achieving the burning rate goal for propellant "B". Conventional methods were used to accomplish this goal, such as increasing ballistic solids and/or fine oxidizer content. The burning rate data are summarized in Table 7. As can be seen from the table, the major increase in burning rate was realized when the 8µ AP (MA) was replaced with uncoated 3.1 UFAP. A result that was initially puzzling was derived from the 86 and 87% solids propellants wherein the solids increase was due to $7-10\mu$ AP (MA). Neither exceeded the burning rate of the 85% solids control formulation even though they showed an internally consistent rate increase. This was explainable, however, in light of later data (Table 7, #AK7365-71) acquired on an identical 86% ballistic solids batch. The mix cycle of this batch was increased by one hour over the former 86% solids batch, resulting in a 47% increase in the burning rate. Apparently the increased mixing time further deagglomerates the UFAP which increases the burning rate.

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(C) FIG. 6. Viscosity Buildup at 120° F on Propellant "A" with (1) \bullet Regular R-45M (AK7591-70) and (2) \blacksquare Modified R-45M (AK7591-76).

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(C) TABLE 7. Effect of Oxidizer Particle Size and Level on Solid Strand Burning Rates of Propellants "A" and "B"

		88	Rallistíc Variables	riables				Burning Rat	Burning Rate r. in./sec.	
							Mix			
Batch	% UFAP	% UFAP	% UFAP	% AP	% AP	% PAP	Cycle,	200	2000	
No. AK-	(0.4µ)ª	e(16.0)	$(3.1_{\mu})^{D}$	(MA)	(UNC)	(UNG)	hrs.	psi	psi	u
7365-33 ^c	50.0	:	:	19.0	:		1.3	1.083	3.026	0.75
7365-35	:	50.0	:	19.0		•	1.3	1.041	2.922	0.74
7365-51 ^d	50.0	:	•	19.0		•	1.1	1.021	2.767	0.72
7365-67	50.0	:	:	20.0	:	•	1.3	0.975	2.844	0.75
7365-69	50.0	:		21.0		•	1.3	1.043	2.974	92.0
7365-71	50.0	:	:	20.0		•	2.1	1.084	3.052	0.75
7365-75	50.0	:	19.0	:	:	•	1.3	1.074	3.148	0.78
7365-77	55.0	:		14.0	:	•	1.6	1.063	3.018	0.75
7365-79	55.0	:	•	:	14.0	:	1.6	0.987	2.856	92.0
7365-81e	51.0	:	:	19.0	•	•	1.6	0.735	2.266	0.82
7365-87 [£]	0.04			20.0	:	10.0	1.6	1.901	6.477	0.878

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Unless otherwise indicated, all batches contain Al-H95 (15%), Silon S (0.5%), Fe₂0₃ (0.5%), with HTPB binder making up remainder. NOIE:

aBDB coated, 0.5%.

b_{Uncoated}.

Control formulation, contains Al-H60 as does Ak-7365-35.

 $^{\rm d}{\rm Fe}_{2}{\rm 0}_{3}$ replaced with ferrocene-formaldehyde polymer, contains Al-H60.

Eburning rate catalysts omitted. $f_{\text{Contains }3\%} \text{ catocene and no Fe}_{203} \text{ or silon s.}$

 $^{
m g}$ Average between 500 and 2000 psia.

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- Increasing the UFAP content at the expense of the $7-10\mu$ AP (MA) also failed to show any increase in burning rate. Again this was understandable, in light of the effect of mix time on burning rate. Replacing the 8µ AP (MA) of this batch with 180µ AP (unground (Ung)) resulted, however, in a 5-6% lowering of the burning rate. This result coupled with the rate increasing effect of the 3.1 µ UFAP underscores the importance of the coarser oxidizer fraction in influencing the burning rate. Strangely enough, though, little effect on burning rate was noted in going from 0.4μ to 0.9μ UFAP. This either reflects the insensitivity of burning rate to UFAP particle sizes less than 1.0µ or indicates the difficulty in breaking up fine UFAP agglomerates in the sub-micron range. This latter explanation is consistent with the observed effect of processing time on burning rate, and significant burning rate increases were later realized with the finer UFAP and long mix cycles. There does appear to be a practical limit to increasing the burning rate by decreasing the particle size of the AP since a burning rate of 3.5 in./sec at 2000 psia was also demonstrated using a 0.55µ UFAP. Evidently sufficient deagglomeration of the UFAP does not take place in this system to distinguish between 0.40 and 0.55µ UFAP.
- (U) Using the binder system and some of the ballistic knowledge developed for propellant "A", a batch of propellant "B" was prepared that nearly achieved the 7.0 in./sec target burning rate (Figure 7). The ballistic modifications consisted of increasing the 0.5µ UFAP by 5%, incorporating a more active PAP and replacing Hycat 6 with catocene which is believed to be more effective (Table 7, #AK7365-87). The attempt here was to achieve the desired burning rate without drastically increasing the propellant sensitivity with higher levels of PAP and Catocene than were used in ANB-3364. By increasing the processing time and making minor ballistic modifications the target burning rate for "B" was achieved as shown in the next section. This illustrates how progress made on propellant "A" was adapted to meeting propellant "B" requirements.

Evaluation and Comparison of Burning Rate Catalysts

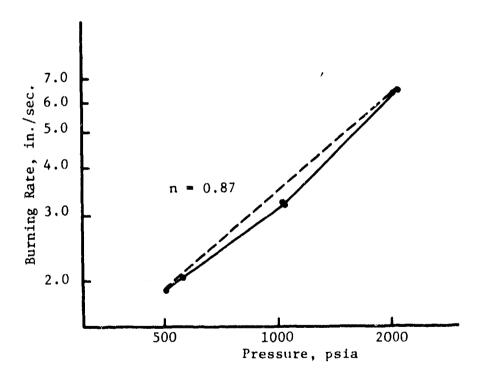
(U) A significant increase in the burning rate of propellant "A" was achieved (Table 2, #AK7591-93, -75) by replacing the pigment grade iron oxide ordinarily used (C. K. Williams, Lot #RY2196) with a red crystalline iron oxide (F. C. Davis Company), a sample of which was provided by the Naval Weapons Center. No other effects, adverse or otherwise, were observed with this new iron oxide; therefore, it was used in all subsequent propellant "A" formulations. This increase brought to three the number of independently achieved burning rate increases to date, the other two being achieved by (1) replacing the 8μ MA AP with 3μ UFAP and (2) increasing the length of the mix cycle (see the previous section). All three factors were combined into one propellant to yield a solid strand burning rate of 3.62 in./sec at 2000 psia (Table 8, #AK7591-1) which exceeded the minimum program goal for propellant "A". An identical batch prepared to confirm this burning rate (Table 8, #AK7591-19), yielded a burning rate of 3.4 in./sec at 2000 psia. The reason for the lower rate is uncertain, but may be a

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Ingredients	<u>wt. %</u>
UFAP (0.39µ) (0.5% BDB) AP (MA) PAP (UNG)	40.00 20.00 10.00
Al-H95 Agerite White	15.00 0.10
Plastinox Catocene	0.10 3.00 1.80
Oronite 6 R-45M (75 eq) DEO (20 eq)	8.90 0.39
TEA (5 eq) IPDI (70 eq)	0.02 0.69
	100.00



(C) FIG. 7. (Upper) Propellant "B" Formulation (Batch No. AK-7365-87) with (lower) Corresponding Plot of Solid Strand Burning Rate vs. Press.

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(C) TABLE 8. Effect of Catalyst and Coarse $\mathrm{NH_4C10_4}$ Fraction on Ballistic Properties

	Mix Cycle (Hrs)	1.5 1.3 1.3 1.1 4.0 4.5
op., 80°F	и	0.82 0.74 0.77 0.77 0.67
Ballistic Prop., 80°F	r ₂₀₀₀ in./sec	2.27 2.86 3.15 3.20 2.77 2.92 3.62e 3.40
	Coarse $\mathrm{NH_4C10_4}$ Fraction at 19%	10µ 10µ 3µ 10µ 5µ 5µ 3µ 3µ
	Burning Rate Catalyst at 1%	None 50/50 Silon S/Fe203 50/50 Silon S/Fe203 50/50 Silon S/Fe203 50/50 Silon S/Ferrocene ^C Ferrocene ^d 50/50 Silon S/Fe203 ^b 50/50 Silon S/Fe203 ^b Fe203
	Batch No. AK-	7365-81a 7365-57 7365-93 7365-93 7365-51 7365-50 7591-19 7591-19

All formulations contain (50%) 0.4 $_{\sqcup}$ UFAP, (15%) Al-H95 and the remainder HTPB binder, unless noted otherwise. NOTE:

 $^{
m a}_{
m Contains}$ (1%) 0.4 $_{
m ii}$ UFAP in place of (1%) burning rate catalyst.

 $^{
m b}$ Red, crystalline Fe $_20_3$.

^cFerrocene-formaldehyde polymer.

 $^{
m d}_{2\%}$ ground ferrocene-formaldehyde polymer and 14% Al-H95.

ench micro strands fired in closed bomb.

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result of different testing methods used. In the former batch, the strands used were exactly 1 inch long and were fired in a closed bomb. The latter batch was tested in regular fashion with standard 5 inch strands.

- (U) It was of interest at this time to learn if the same burning rate achieved with the 50/50 mixture of Silon S and the red crystalline iron oxide could be achieved or surpassed by using this new iron oxide as the sole burning rate catalyst at the same total percent. The rate achieved (Table 8, #AK7591-5A) did not equal the rate (Table 8, #AK7365-93) achieved by the 50/50 combination of Silon S and the same crystalline iron oxide. Λ synergistic effect is indicated by this and data from another program regarding the effect of Silon S concentration.
- (U) To determine just how much the catalyst was contributing to the burning rate, a batch was prepared in which the 0.4 μ UFAP was increased by 1%, replacing the catalyst (Table 8, #AK7365-81). The data indicate that the catalyst system contributes approximately 25% to the burning rate at 2000 psia. However, due to a higher slope, 0.82 versus 0.77 for the catalyzed propellant, the contribution increases with decreasing pressure.
- As a possible replacement for Fe₂0₃, a solid ferroceneformaldehyde polymer was evaluated with Silon S (Table 8, #AK7365-51). A lower burning rate and pressure exponent (n) resulted. It appears this ferrocene polymer does not dissolve to any appreciable extent in the binder, solution being important if ferrocene compounds are to be effective burning rate catalysts. To offset this lack of solubility the ferrocene polymer was ground to a very fine powder and evaluated at the 2% level (Table 8, #AK7591-50) to see if it would now approximate the behavior of soluble ferrocene derivatives, i.e., show a nearly linear increase with level. Not only did it not significantly increase burning rate, but it severely curtailed processability and promoted a very rapid cure. A possible explanation for its poor behavior as a burning rate catalyst is its high polymeric nature which reduces its ability to interact with the NH4ClO4, contrary to the low molecular weight liquid and solid ferrocene derivatives, such as catocene and n-butyl ferrocene.
- Fe $_20_3$ to be used in propellant "A", a comparison of activity of the sample of crystalline Fe $_20_3$ (Lot 15) from NWC was made with a 10 lb lot of crystalline Fe $_20_3$ purchased from Frank C. Davis Company to see if lot-to-lot variations existed. No differences in burning rate were observed (Table 9, #AK7591-48, -58), but the batch containing the (Lot 15) Fe $_20_3$ did process somewhat better. A significant increase in burning rate (Table 9, #AK7591-60) was achieved, though by grinding a portion of the crystalline Fe $_20_3$ from the 10 lb lot for two hours in Freon using Al $_20_3$ 1/4 inch cylinders for grinding, and a mechanical paint shaker to provide agitation. The resulting powder was much finer than the unground material and was redder in appearance; the average particle size was not determined. The finer Fe $_20_3$ also appeared to function as a better cure catalyst since the data show a shorter potlife for this batch.

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Effect of Catalyst Variations and UFAP Level on Processing, Ballistic and Mechanical Properties TABLE 9. (O)

	Oxidizer	zer	Ballistic	Ballistic Prop.,80°F	Mech. Prop., 77°F	rop., 7	7°F		
Batch No. AK-7591-	% UFAP (0.55µ)	% UFAP (5μ)	r2000 in./sec	Pressure Exponent n	σm, psi	ε m,	Eo, psi	Potlife ^a Hrs	Shore "A"
AK7365-81 ^b	51.00	19.00	2.27	0.82	•	:	•	•	:
48 ^c	50.00	19.00	3.40	0.76	0.96	16.6	919	1.0	9
58 ^d	50.00	19.00	3.40	0.77	118	19.8	029	0.7	99
₉ 09	50.00	19.00	3.56	0.79	124	18.1	772	0.5	63
68 ^{e, f}	45.00	24.00	3.47	0.77	73.2	25.1	344	1.0	37
70e,f	40.00	29.00	3.40	0.75	9.92	24.1	353	2.0	44

0.5% Fe₂0₃, 15% binder [(0.2% aging stabilizers, All batches contain 15% H-95 Al, 0.5% Silon S, 0.5% Fe₂0₃, 15% binder [(0.2% aging stabilize) (4.8%) Oronite 6, (85 eq.) R-45M, (10 eq.) DEO, (5 eq.) TEA, (70 eq.) IPDI] unless otherwise NOTE:

 $^{\rm a}{
m Time}$ to 50 kpoise at 120°F and 10,000 dynes/cm $^{\rm 2}$ shear stress.

^bContains (1%) 0.4μUFAP in place of 1% burning rate catalyst.

^CContains new lot crystalline Fe $_20_3$ received from NWC.

 $^{
m d}_{
m Contains}$ new 10# lot of crystalline Fe $_2$ 0 $_3$ purchased from Frank C. Davis Co.

 $^{
m e}_{
m Contains}$ new 10 % lot of crystalline Fe $_20_3$ ground 2 hrs. on a paint shaker.

 $f_{\rm Contains}$ (65 eq.) IPDI, (4.5%) Oronite 6, (0.5%) aging stabilizers

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- (U) Since the ground Fe $_2$ 0 $_3$ gave a faster burning rate than asreceived, an evaluation was made to determine if the concentration of 0.55 $_\mu$ UFAP could be reduced and still meet the 3.5 in./sec, burning rate goal at 2000 psia. The data (Table 9, #AK7591-68, -70) indicate that a drop of 5 wt% in this fine oxidizer very nearly meets the desired burning rate. Also, the processability improves very rapidly as the level of 0.55 $_\mu$ UFAP is lowered. The binder was altered for these batches to provide a softer propellant, which explains the lower tensile and higher elongation values.
- (U) Utilizing the information gained in developing propellant "A", a batch of propellant "B" was prepared (Table 10, #AK7591-54) that exceeded the 7.0 in./sec burning rate goal at 2000 psia using four percent Catocene. Additionally, better mechanical properties were realized than before, but the Shore "A" hardness was too high and the potlife was not as long as desired. It was expected that the potlife would be significantly extended by the use of modified R-45M, but as stated earlier, no improvement in potlife was realized until 2,4-pentanedione was incorporated in the formulation.
- (C) Although the burning rate goal at 2000 psia for propellant "B" was exceeded using 4% Catocene and 8% unground PAP, a series of batches were prepared in which various combinations of oxidizer blend and Catocene level were evaluated to determine how low in Catocene and UFAP level the desired burning rate could be achieved (Table 10). This was desirable from the standpoint of improving the processing and safety characteristics of this propellant. The study revealed that the catalyst level was significantly more effective than the 0.55 μ UFAP level in raising the burning rate. Also, to improve processing the coarse NH₄ClO₄ fraction was changed from 5 μ to 10 μ average particle size with a small loss in burning rate. The final choice in Catocene level was 4% since hazard tests showed little change in safety characteristics in going from the 3 to 4% level, thus allowing improved processability.

Evaluation of Alternate Metal Fuels, Oxidizer and Fluorocarbon Plasticizer

- (U) A number of ballistic modifications were evaluated that had shown ballistic improvements in other propellant systems at Aerojet Solid Propulsion Co. It was hoped that they might do so with the propellant "A" formulation, but no advantages were observed and in one case processing was severely degraded. These modifications are discussed in the following paragraphs.
- (U) Magnesium and Magnesium/Aluminum Alloy Powders. The replacement of aluminum with magnesium powder and a powder consisting of a 65/35 magnesium/aluminum alloy showed no improvement in burning rate (Table 11, #AK7591-5B, 5C, 5D). The failure, however, of magnesium to elevate the burning rate was not too surprising, since burning rate increases observed with this metal powder were in propellants containing varying amounts of fluorocarbons. The magnesium powder did not adversely affect processing, cure or mechanical properties.

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(C) TABLE 10. Effect of Catocene Level and Oxidizer Particle Size on Ballistic Properties of Propellant "B"

	Shore "A" Hardness	72	45	32	30	70	67
	Potlife ^a (Hrs)	0.5	•	8.0	1.0	•	÷
rop.	E _o , psi	901	433	307	240	1074	781
ical P	е п,	20.0 901	74.6 22.6	60.8 28.4	30.3	16.1 1074	16.7
Mechanical Prop.	om, psi	145	74.6	8.09	53.1	149	115
Prop.	qu	0.84	0.85	0.85	0.84	98.0	0.80
Ballistic Prop.	r ₂₀₀₀ in./sec	7.30	6.40	6.55	07.9	86.9	6.78
ables	Catocene %	0.4	3.0	3.0	3.0	3.5	4.0
Ballistic Variables	% UFAP (5μ)	17.00	12.00	17.00	22.00	17.00	22.00
Ballis	% UFAP (0.55µ)	45.00	50.00	45.00	70.09	45.00	40.00
	Batch No. AK-7591-	54	99	72	74	87	68

All batches contain (8%) Ung PAP, (15%) Al H-95, and remainder R-45M/IPDI binder, unless otherwise noted. Mix cycles are 4 hours. NOTE:

 $^{\rm a}_{
m Time}$ to 50 kpoise at 120"F and 10,000 dynes/cm $^{\rm 2}$ shear stress.

 $^{
m b}_{
m Average}$ between 500 and 2000 psia.

Cused R-45M-25 in place of R-45M.

 $^{
m d}_{
m Use}$ (MA) NH $_{
m 4}$ ClO $_{
m 4}$ in place of (5 $_{
m \mu}$) UFAP.

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(C) TABLE 11. Effect of Ballistic Variations on Burning Rate of Propellant "A"

	le, ^r 2000 in./sec	2.27	3.20	3.15	3.14°	3.14 ^c	3.40	3.03
	Mix Cycle, hrs.	1.6	1.5	2.5	2.5	2.5	4.5	1.6
	Coarse AP at 19%	(5h)	(10 _µ)	(3h)	(3 ⁿ)	(3 ^r)	(3µ)	(10h)
Ballistic Variables	Metal Powder at 15%	A1-H95	A1-H95	Mg/Al alloy (65/35)	Ng	Al Class II	A1-H95	A1-H95
Ballis	Burning rate catalyst at 1%	None	50/50 Silon S/Fe ₂ 0 ₃	50/50 Silon S/Fe ₂ 0 ₃	50/50 Silon S/Fe ₂ 0 ₃ ^b	50/50 Silon S/Fe ₂ 0 ₃	50/50 Silon S/Fe ₂ 0 ₃	$50/50$ Silon $8/\text{Fe}_20_3$
	Batch No. AK-	7365-81 ^a	7365-93	7591-5B	7591-5C	7591-5D	7591-19	7365-89 ^d

NOTE: All formulations contained 50% UFAP (0.4 μ) and 15% R-45-IPDI binder.

^aControl batch. Contains (1%) 0.4 μ UFAP in place of 1% burning rate catalyst.

bRed crystalline iron oxide.

Small solid strands fired in a closed bomb.

d_Contains (45%) 0.4 μ UFAP and (5%) unground methylamine perchlorate (MAP).

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- (U) <u>Methylamine Perchlorate (MAP)</u>. A portion of the 0.4μ UFAP was replaced with a like amount of unground MAP to see if a significant increase in burning rate could be achieved. The burning rate achieved (Table 11, #AK7365-89), however, was not quite as good as that for the control (Table 11, #AK7365-93).
- (U) Fluorocarbon Plasticizer. An attempt was made to incorporate a fluorocarbon ester into propellant "A" and evaluate its effect on burning rate. This ester was prepared by condensing 216 gms (0.5 mole) of 1,1,9-trihydrohexadecafluoro-1-nonanol with 144 gms (1.0 mole) of 2-ethylhexanoic acid catalyzed with one gm of conc. H₂SO₄ all in 250 ml of toluene. The solution was refluxed for 6 hrs in which time nine ml of water were azeotroped off by the toluene signifying completion of the reaction. The toluene solution was washed twice with distilled water dried over anhydrous MgSO4 and filtered to remove drying agent. The toluene was removed under vacuum on a rotary film evaporator and the resultant oil was distilled under vacuum to yield ~200 gms of the desired ester boiling at 107-109°C/3mm Hg. Only half of the Oronite 6 was replaced with this material, but it made the propellant difficult to process resulting in a brittle propellant which could not be tested for burning rate. Since it appeared that significant amounts of fluorine could not be incorporated into this propellant system without severely degrading processing and mechanical properties, no further effort in this direction was planned.

LINER/PROPELLANT BOND STUDIES

- (U) One of the problem areas identified in the 1969 program was the poor bonding between the propellant and the liner which was suspected to be the cause of motor failures. Therefore, motor tests were conducted early in the current program to demonstrate that successful tests could be made. In preparation for an early motor demonstration test, promising liners developed on another program were evaluated with representative propellants of this program.
- (U) To measure liner propellant bond, 434-4 liner, both partially cured and completely cured prior to casting, as well as SD-898 were evaluated (Table 12). It is noteworthy that the excellent propellant-liner bond was achieved with the 434-4 liner in light of the fact that good bonding to HTPB propellant has been, heretofore, difficult to achieve, especially with UFAP containing propellants.
- (U) DPT molds were prepared with each candidate propellant using a modified 434-4 liner. Previous tests had shown 434-4 liner to give good bonds to these propellants. The modified 434-4 liner also gave good bonds to these propellants as shown on page 33.

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(U) TABLE 12. DPT Liner Evaluation Using 86% Solids a 10-1b Batch of Propellant "A".

Liner	Cure state	Tensile, psi	Type of failure
SD-898	Cured	50.8	Adhesive
434-4	Cured	52.5	Adhesive
434-4	Partially cured ^b	92.8	Cohesive
	Liner	Formulations	
		SD-898	

Liner rorm	ITACIONS	
Ingredients	SD-898 (wt.%)	
Sb ₂ 0 ₃	9.50	
P-33	9.50	
Refracil	6.00	
Agerite White	2.00	
Plastinox 711	1.00	
Thixcin E	1.00	
ZrAA	1.00	
Cr Oleate	0.50	
HC-434	63.52	
DER-332	5.98	
	100.00	

a_{Batch No. AK-7365-73}.

bCured 4 hrs. @ 135°F.

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DPT Values for Candidate Propellants Using Modified 434-4 Liner at 74.6°F

	ANB-3394 (10GP-2213)	ANB-3395 (10GP-2248)
Tensile, psi	68.9	94.9
Type of Failure	In Propellant	In Propellant

Liner modification consisted simply in reducing the filler concentrations to improve the viscosity and flow properties of the 434-4 liner. No changes were made in the polymer composition.

(U) Since this modified liner appeared to be satisfactory, it was used as the liner for the propellant grains that were prepared and delivered to Naval Weapons Center as per contractual requirements.

HAZARD TESTING OF PROPELLANTS "A" AND "B"

(U) Safety data acquired on the propellant "B" formulation containing four percent Catocene indicated this propellant had high friction sensitivity. At three percent Catocene the friction sensitivity was quite low indicating a less hazardous propellant. Unfortunately, the more sensitive propellant also had a high Shore hardness while the less sensitive one had a low hardness reading, and since friction sensitivity tends to go up with propellant hardness it was not known whether high catocene, high Shore hardness or both were responsible for the increased friction sensitivity. Since subsequent batches at four percent Catocene and lower Shore "A" hardness values did not show high friction sensitivity it was concluded that hardness level was responsible.

Halards Test Data

Propellant "B"

Batch No.	ΛΚ7591-54 (See Table 10)	AK7591-74 (See Table 10)
Bureau of Mines Impact		
50% Fire Point	11 cm/2 kgm	• • •
Friction (Rotary)		
Gms load/RPM	400/3000	2000/3000
DTA		
Onset of Exotherm	328°F	• • •
Exothermic Peak	380°F 423°F	• • •
Autoignition	423 ľ	• • •

(U) In selecting the final composition for propellant "B", consideration was given to the hazard characteristics of the two compositions from which the candidate formulation was selected. The desired composition from the standpoint of processing, Λ K7591-89, contained 40% 0.5 μ UFAP and 4% Catocene and it was feared that the

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higher Catocene level of the former would give a less safe propellant. The data shown below, however, indicated little or no differences between the two, allowing the desired choice to be made based on processability. In addition, hazard tests were run on the uncured propellants to see if any special processing or handling techniques were required. The data indicated both uncured propellants were quite safe to handle in ordinary fashion.

Hazard Tests on Propellant "B" Uncured and Cured

		MK7591-87 able 10) Uncured	Batch A (See Ta Cured	K7591-89 ble 10) Uncured
Bureau of Mines Impact				
50% Fire Point, cm/2kg	10.0	18.7	11.4	19.2
Friction, Rotary	2200	2000	2200	2000
Gms load/RPM	2300 3000	3000 5200	<u>2200</u> 3000	<u>3700</u> 4700
DTA				
Onset of Exotherm Exothermic Peak, °F	329 395	•••	309	• • •
Ignition, °F	444	• • •	432	• • •

(U) Both candidate formulations from 10-1b batches were again checked for impact and friction sensitivity in the uncured state to determine possible differences in hazard properties due to scale-up. None were evident from the data.

Safety Tests on Both Uncured Candidate Propellants

	ANB-3394 (10GP-2194)	ANB-3395 (10GP-2220)
Bureau of Mines Impact		
50% Fire Point	26 cm/2kg	14.4
Rotary Friction		
gm 1oad/3000 RPM	2420	3200

(U) I.C.C. hazard classification tests were run on ANB-3394 and -3395 as outlined in TB-700-2. The results (Table 13) show both propellants to be Class $^{11}B^{11}$ explosives.

SELECTION OF CANDIDATE FORMULATIONS

(U) In selecting the candidate formulations for this program the following criteria were used in decreasing order of importance: (1) burning rate, (2) processing characteristics, (3) mechanical properties and (4) hazard characteristics.

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(U) TABLE 13. Safety Data, NWC Candidate Propellants

		Candidat	es
		A (ANB-3394)	B (ANB-3395)
1.	Bureau of Mines Impact 50% pt., cm/2 Kg. wt.	17.0	13.5
2.	DTA Results		
	Onset of Exotherm, °F Exotherm Peaks, °F Ignition, °F	335 435 451	328 385 428
3.	Copper Block		
	Autoignition, °F	433	368
4.	Thermal Stability at 75°C for 48 hr.	No change	No change
5.	Detonability with No. 8 Blasting Cap	Burned in 8 sec.	Burned in 4 sec.
6.	Unconfined Burning for 2 in. cube	9 sec.	4 sec.
7.	Friction Rotary, 50% point	1750 gm. load 3000 rpm	1700 gm. load, 3000 rpm
8.	Spark Gap Test, 50% pt.	3.30 joules	2.70 toules

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Candidate "A" (ANB-3394)

- (U) The final formulation selected for candidate "A" (ANB-3394) is shown in Table 14. The extent to which this formulation satisfied the selection criteria is summarized below:
- (U) Burning Rate. The desired goal was 3.5-4.0 in./sec at 2000 psia. This formulation achieves the low end of this range. Although rates higher than 3.5 in./sec have been achieved, too great a sacrifice in processing properties was required to make those compositions feasible.
- (U) Processing Characteristics. A potlife of at least three hours at 120°F was required to successfully scale-up this formulation and cast motor grains. With the use of modified R-45M this minimum potlife was realized. This formulation had virtually no static flow characteristics, but under suitable vibration flow was adequate to cast void-free grains.
- (U) Mechanical Properties. Tensile strengths of 100-150 psi and elongations of 30% were desired. This formulation was low on both properties; however, it represented an adequate compromise between formulations that achieved the desired tensile strengths but were very low on elongation, and vice versa.
- (U) <u>Hazard Characteristics</u>. A formulation was desired that would qualify as a Class "B" explosive. This formulation was classified as such by I.C.C. tests.

Candidate "B" (ANB-3395-1)

- (U) The final formulation selected for candidate "B" (ANB-3395-1 is shown in Table 14. The extent to which this formulation also satisfied the selection criteria is summarized below:
- (C) Burning Rate. A burning rate of 7.0 in./sec or higher at 2000 psia was sought for this formulation. The rate achieved, however, is about 4-5% low for this candidate formulation. Rates greater than 7.0 in./sec were achieved in other compositions, but again, as in ANB-3394, the sacrifices in processing characteristics were too great to make these compositions feasible. Also, the overall pressure exponents were too high in some cases.
- (U) Processing Characteristics. As in ANB-3394, a potlife of at least three hours at $120\,^{\circ}\text{F}$ was required to successfully scale-up this formulation and cast motor grains. This minimum potlife was achieved with the use of modified R-45M and HAA. This formulation also exhibited static flow and under vibration was more than adequate to cast void-free grains.
- (U) Mechanical Properties. Mechanical property requirements were the same as for ANB-3394. This formulation also fell short of the minimum desired properties, but more nearly achieved them than did ANB-3394.

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(C) TABLE 14. Candidate Formulations, Propellants "A" and "B".

Designation	A (ANB-3394)	B(ANB-3395-1)
Ingredients	wt. %	wt. %
NH ₄ C1O ₄ (0.5μ)	45.00	42.00
NH_4^{C10} (5 μ)	24.00	
NH_4C10_4 (10 μ)		20.00
NH ₄ C10 ₄ (Porous, 180μ)		8.00
A1-H60	15.00	15.00
Catocene		4.00
Fe ₂ 0 ₃ (crystalline)	0.50	
Silon S	0.50	
Agerite White	0.20	0.20
Plastinox #711	0.30	0.30
НАА		0.20
Oronite 6	4.50	0.30
R-45M-35 (78 eq.)	9.08	9.08
DEO (15 eq.)	0.21	0.21
TEA (7 eq.)	0.02	0.02
IPDI (100 eq.)	0.69	0.69
	***************************************	Community of the Control of the Cont
	100.00	100.00

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(U) <u>Hazard Characteristics</u>. This formulation was expected to qualify as a Class "B" explosive. I.C.C. tests confirmed this classification.

PROPELLANT SCALE-UP AND GRAIN PREPARATIONS

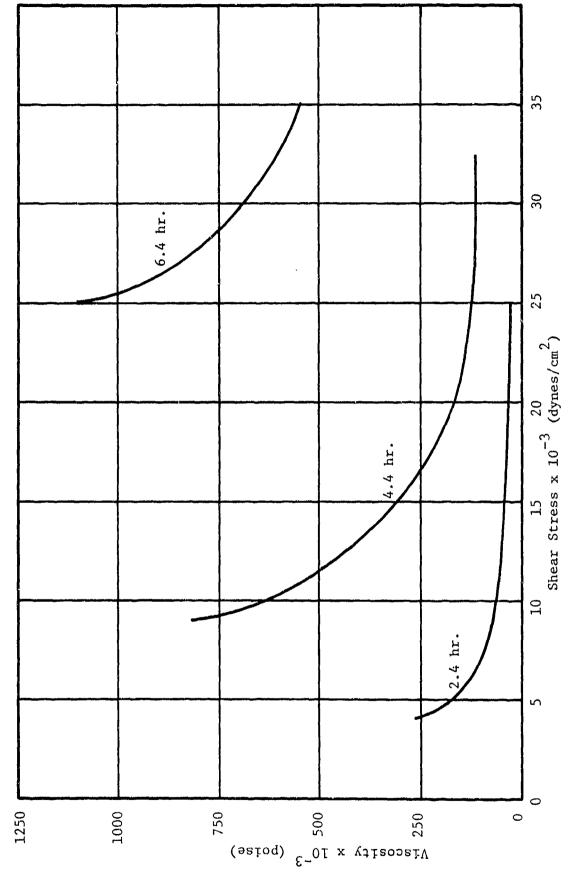
- Early in this program an 86% solids version of propellant "A" was scaled up to a 10-1b batch size (Table 15) to determine scaleup problems, if any, and to obtain viscosity buildup, liner bonding and grain casting data. The batch mixed well, using a mix cycle one hour longer than had been used in the 300 gm batch studies, and was vacuum castable. However, the material flowed only with vibration and had a very short potlife (Figure 8). Ballistic and mechanical property data were measured on this formulation, showing it to have adequate mechanical properties, but falling about 13% low in achieving the desired burning rate (Table 16). Subsequent 10-1b batches were not prepared until the candidate formulations, ANB-3394 and ANB-3395 (ANB-3395-1 was a later modification involving the addition of HAA) were selected. Three 10-1b batches of each candidate formulation were prepared to provide sufficient propellant to cast 28 phenolic sleeves, 2.00 in. dia x 7.00 in. long. These sleeves were lined with modified 434-4 liner that was precured for 1.5 hours at 75°C prior to casting with propellant. Final cure of the liner took place with the propellant cure after it was cast, which resulted in excellent liner/propellant bonds.
- Both candidate propellants were mixed without difficulty, however ANB-3394 had noticeably better potlife than ANB-3395. Viscosity buildup data (Figure 9) also indicated that ANB-3395 had far less potlife than ANB-3394, due to the catalytic effect of catocene accelerating the cure rate. This problem was later resolved with the addition of HAA (0.2%) to the formulation, thereby changing the designation to ANB-3395-1. Mechanical properties were measured on both candidate formulations over the temperature range of 160 to -40°F (Table 17) showing good agreement within each three batch set as well as very little variation in elongation over the entire temperature range. However, nearly all the grains cast contained voids as revealed by X-radiography, requiring the preparation of additional 10-1b batches to prepare sufficient good grains for delivery to NWC, China Lake. This time, however, casting procedures were modified to assure a higher yield of good grains, The modifications included: (1) overcast additional 1-inch, (2) low frequency - medium amplitude cast vibration and (3) small stream casting. These changes resulted in a much higher yield of void-free grains, sufficient in number to meet contractual requirements.
- (U) The scale-up of ANB-3394 and ANB-3395-1 to 175-1b batch size to cast the 5-in. dia x 15-in. long grains, was accomplished essentially without problem. All grains and cartons cast were shown to be void-free by X-radiography. This success was largely attributed to the availability and use of casting equipment that allowed all the units to be kept under vacuum throughout the whole casting operation and beyond, while being subjected to low frequency medium amplitude vibration.

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(C) TABLE 15. 86% Solids - 10-1b Scale-Up Batch Of Propellant "A"a

Ingredients	Weight %
UFAP (0.39μ)(0.5% BDB)	50.00
AP (MA)	20.00
A1-H95	15.00
Fe ₂ 0 ₃	0.50
Silon S	0.50
Agerite White	0.10
Plastinox 711	0.10
Oronite 6	4.80
R-45 M (75 eq)	8.01
DEO (20 eq)	0.35
TEA (5 eq)	0.02
IPDI (70 eq)	0.62
	100.00

 $^{^{\}mathrm{a}}$ The TEA was added after IPDI in the mix cycle.



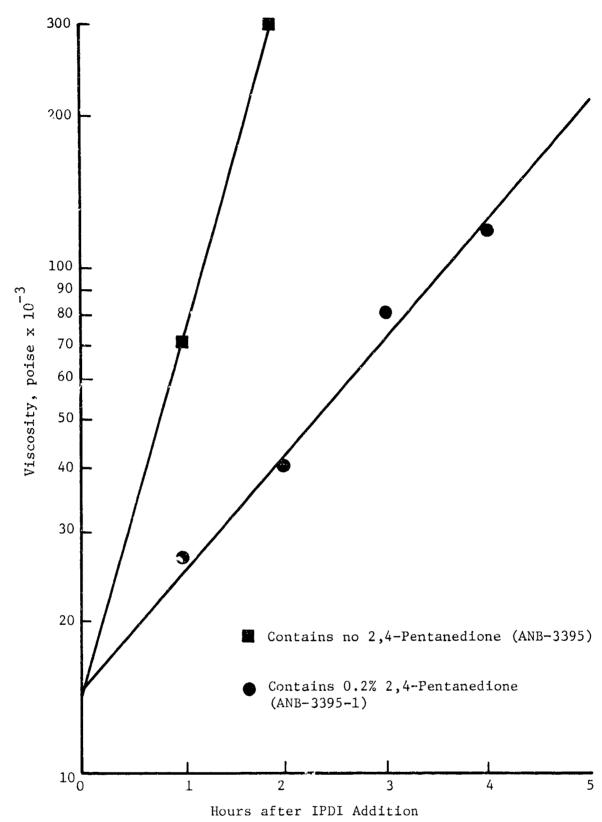
(U) FIG. 8, Viscosity vs. Shear Stress at 110° F Taken at Two-Hour intervals^a for 10-1b Batch 0f Propellant "A" (Batch # AK 7365-73). (a) Time taken from IPDI addition.

(U) TABLE 16. Processing, Mechanical and Ballistic Properties of 86% Solids Formulation a for Propellant "A"

	Processing Properties		
	2.4 hrs	4.4 hrs	6.4 hrs
Viscosity buildup, 110°F, at infinite shear (kpoise), hrs after IPDI addition	1.7	55	7.1
	Mechanical Properties		
	160°F	77°F	-40°F
$\sigma_{ m m}$, psi	78.0	129.0	227.3
ε, %	16.1	18.3	17.0
, c P , s , s , s , s , s , s , s , s , s ,	16.7	18.9	17.3
E _o , psi	495	760	1829
	Ballistic Properties		
	500 psia	1000 psia	2000 psia
$R_{ m B}$, in./sec	1.084	1.884	3.052
п	0.75		

 a One-1b batch no. AK-7365-71.

 $^{^{}m b}$ Determined on ten-1b batch no. AK-7365-73 of identical formulation to AK-7365-71.



(U) FIG. 9. Viscosity Build-up of ANB-3395 (B) at $120^{\circ}F$ and 10,000 dynes/cm² Shear Stress with and without 2,4-Pentanedione.

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TABLE 17. Mechanical and Ballistic Properties of ANB-3394 ("A") and ANB-3395 ("B") <u>(a)</u>

		Mech	Mechanical Properties	perties		Balli	Ballistic Properties, 80°F	rties, 80°	F	
Batch No. 10GP-	Shore	Temp.,	om, psi	е ^ш 3	E _o , psi	r ₅₀₀ in/sec	r ₁₀₀₀ in/sec	^r 1500 in/sec	r ₂₀₀₀ in/sec	u
2194 ^a	28	77	68.2	25.1	337	1.21	2.06	i	3.43	0.75
2212 ^a	31	2.2	59.7	17.9	395	1.20	2.04	I	3.45	0.76
2213 ^a	40	77	78.2	18.5	488	1.20	2.02	2.74	3.40	0.74
2213	-	160	47.2	14.0	358	ŀ	ļ	1	ļ	ļ
2213	1	0	150.3	19.4	1029	1		ł	!	!
2213	!	05-	327.1	17.7	2419	}		1	1	1
2220 ^b	48	77	84.1	17.5	609	2.19	3.58	1	6.50	0.86
2228 ^b	47	77	76.7	15.3	586	2.27	3.55	1	09.9	0.88
2248 ^D	48	7.7	85.6	16.8	637	2.17	3.54	5.15	6.70	06.0
2248	1	760	61.7	16.4	433	-		ŀ	-	!
2248		0	197.7	16.9	1535	[1		1	ļ
2248		-40	445.2	14.0	4192	!	ľ	!	1	1

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a Candidate "A" propellant.
b Candidate "B" propellant.

^c Measured in pressure range 1000 - 2000 psi.

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Additionally, multiple small stream casting was used to enable sufficient deaeration to take place.

STRAIN MEASUREMENTS ON CANDIDATE PROPELLANTS

Bore strain measurements were made on both ANB-3394 and -3395-1 candidate formulations. Two sets of four 2 x 3-in, steel strain cylinders were lined with modified 434-4 liner. Each set of four cylinders was fitted with cores having the diameters 0.5, 0.6, 0.7 and 0.8 inches and cast with a candidate propellant. Since 135°F was the cure temperature for both sets, this temperature was taken as the zero strain temperature. All the strain cylinders were then lowered to -40°F in discreet temperature intervals for fixed time periods, and the change in bore diameter was measured at each temperature. These data were used to measure bore strains. If the propellants survived this cycling down to -40° F, they were returned to room temperature and then cycled directly down to -40 $^{\circ}$ F. This cycle was repeated five more times or until propellant failure occurred, whichever came first. ANB-3395-1 survived all the cycles at all the strain levels, but ANB-3394 failed during the first cycle (Table 18). Inspection of the failure cracks along the bore revealed that they propagated axially rather than longitudinally as is usually the case. They also were found to originate from subsurface voids generated during the casting of these cylinders, as a result of poor propellant flow characteristics coupled with reduced casting volume due to the presence of the cores. It is expected, but has not been demonstrated on this program that grains of ANB-3394 would show greater strain capabilities if void free.

SMALL MOTOR TEST FIRINGS

- (U) Initial test firings of ANB-3394 and ANB-3395 in 2-in.-dia x 6.25-in.-long end burning grains resulted in erratic pressure-time traces (Figures 10 and 11). Initial deposition of condensable material on the nozzle was blamed for these results and subsequent firings (Figures 12 and 13) used contoured boron nitride nozzles. Boron nitride nozzles have shown more resistance to deposition than carbon nozzles and confouring was expected to provide smoother gas flow.
- (U) As can be seen from the pressure-time traces, going to contoured boron nitride nozzles showed some improvement but did not adequately solve the deposition problem. It appears that the coarse H-95 aluminum is not completely burned in the initial phase of the firing, which in combination with a low motor L* and an unfavorable aft-closure design, causes deposition of condensable species in the exhaust. The shape of the pressure-time (p-t) trace and level of the equilibrium pressure of these firings, however, indicate that grain integrity is not a contributing factor to the pressure irregularities. The problem of low L* and consequently low residence time is frequently encountered when high r propellant is test fired in motors designed for low r propellants. The average pressures were calculated from these pressure-time traces and the resulting burning rates were in good agreement

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(U) TABLE 18. Results of Strain Measurements

ANB-3394

Strain Cylinder, no.	1	2	3	4
Bore Diameter, in. Cycles Completed Failure Temp., °F % Strain at Failure Cause of Failure	0.50	0.60	0.70	0.80
	None	None	None	None
	40	40	20	0
	7.2	8.9	8.3	7.1
	Void	Void	Void	Void

ANB-3395-1

Strain Cylinder, no.	1	2	3	4
Bore Diameter, in. Cycles completed Failure Temp., °F % Strain at Failure Cause of Failure	0.50 1 -40 18.7 ESC ^b	0.60 2 -40 15.6 ESC ^b	0.70 2 -40 11.8 ESC ^b	0.80 6 9.4

a b Exceeded strain capabilities.

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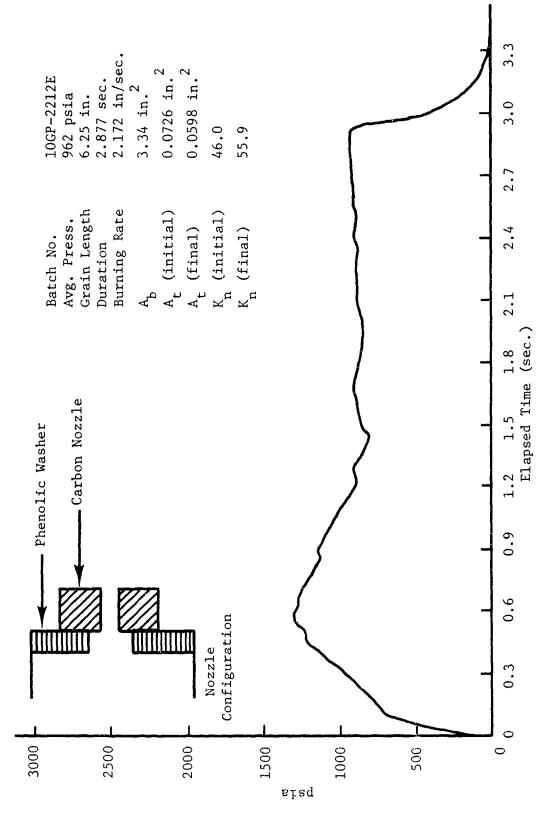
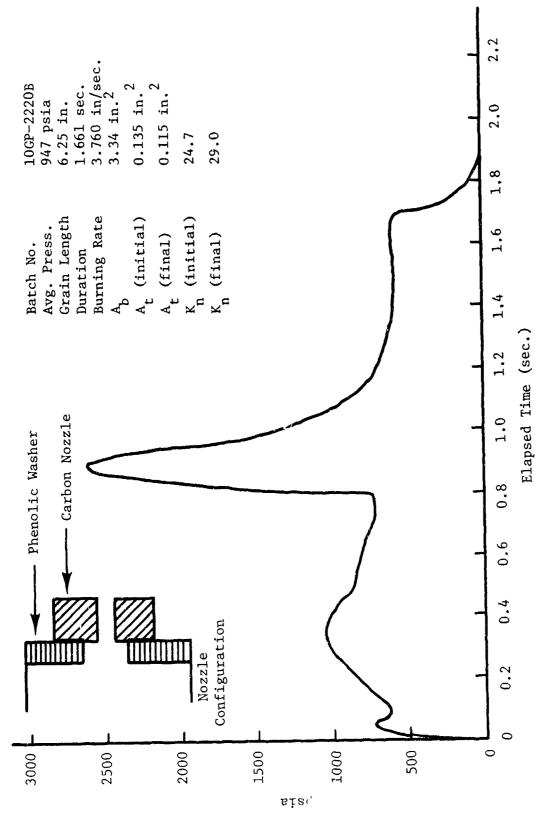


FIG. 10. Pressure-Time Trace of ANB-3394 (A) Motor Firing Using a Non-Contoured Carbon Nozzle (n)

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Pressure-Time Trace of ANB-3395 (B) Motor Firings Using a Non-Contoured Carbon Nozzle FIG. 11. (E)

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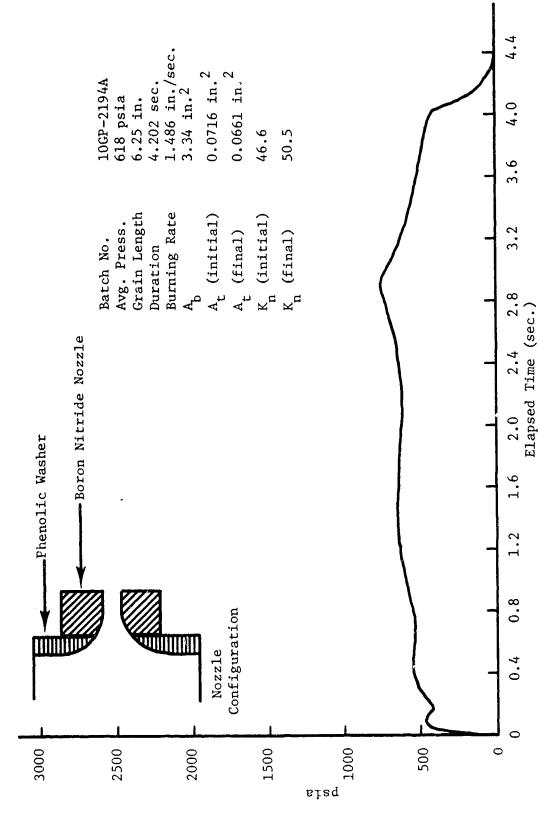


FIG. 12, Pressure-Time Trace of ANB-3394 (A) Motor Firing Using a Contoured Boron Nitride Nozzle. (E)

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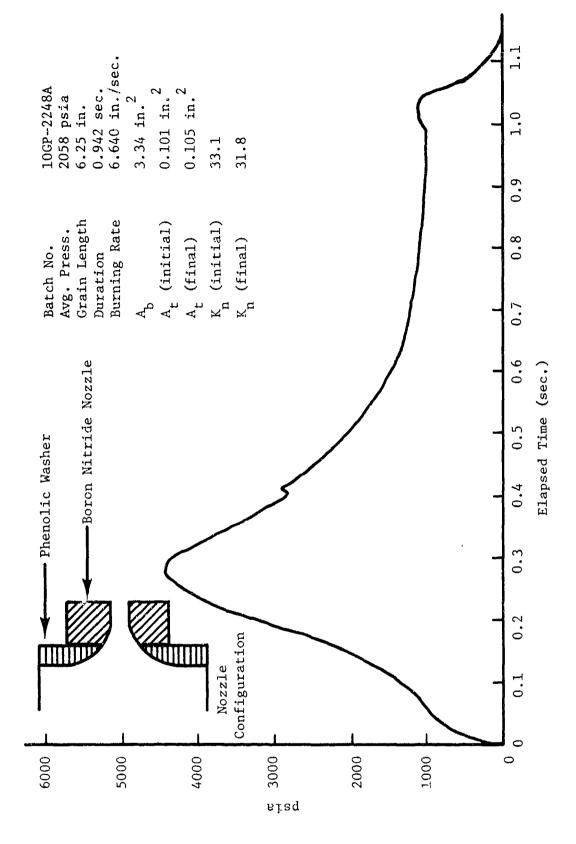


FIG. 13. Pressure-Time Trace of ANB-3395 (B) Motor Firing Using a Contoured Boron Nitride Nozzle. <u>e</u>

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with the solid strand rates from the same propellants (Figure 14).

Subsequent batches were prepared with H-60 aluminum replacing the coarser H-95 aluminum. This was expected to improve the combustion efficiency of the aluminum and thereby reduce deposition. To increase L*, firings were made using 3-in. grains. Also, 0.25 in. of a nonaluminized propellant was cast on top of these grains that served to preheat the nozzle prior to ignition of the candidate propellants. Preheating the nozzle was expected to further minimize deposition and ignition of the main grain was signaled by a sharp rise in pressure. All these grains (Figures 15-17) exhibited successful motor firings. Some of the grains exhibited progressive pressure-time traces which could have been a result of uneven ignition of the main grain by the prewarming grain. Interestingly, one motor fired successfully without the benefit of the prewarming grain (Figure 18), but postfiring inspection of the motors disclosed slag on the nozzle that was not present when the prewarming grain was used. This was interpreted as a positive contribution by these grains towards minimizing deposition.

SUMMARY OF PROPERTIES AND AGING RESULTS FOR ANB-3394 AND ANB-3395-1

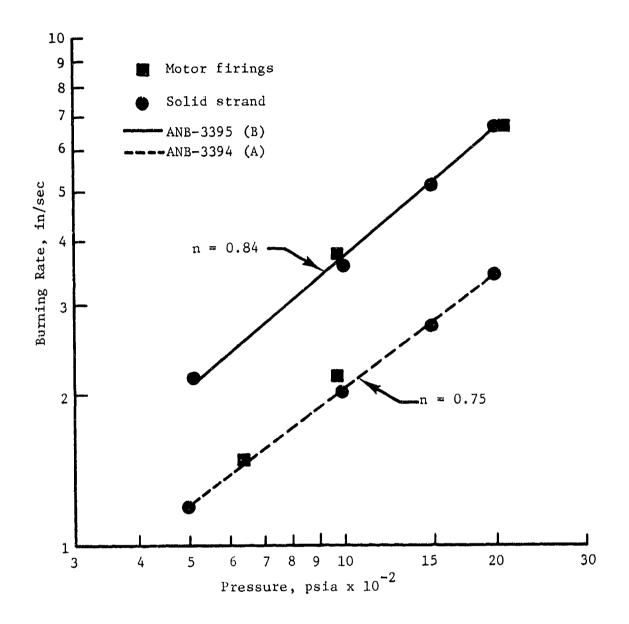
- (U) The processing, mechanical, ballistic and liner/propellant bond properties are summarized for ANB-3394 and ANB-3395-1 in Tables 19 and 20, respectively. In addition, the results of one month aging at 135°F on these properties are also presented in these tables. The major changes brought on by this short aging period were chiefly in the mechanical properties. Both formulations showed increased Shore "A" hardness readings which were probably a result of incomplete cure at the time of initial property measurements, progressing to final cure during aging.
- (U) The liner/propellant bond properties only showed improvement on aging with failure occurring in the propellant, and the ballistic properties essentially remained unchanged. Solid strand and motor burning rates are presented in Figure 19 for both candidate formulations and show excellent agreement with each other.

RECOMMENDATIONS FOR FUTURE WORK

(U) We recommend that additional work be initiated to improve the potlife and mechanical properties of both propellants. An approach to improving potlife would be an evaluation of the cure catalytic effect of the coated UFAP. A coating without catalytic properties should greatly increase potlife. Further, mechanical properties could be improved by lowering or eliminating plasticizer if sufficient increase in potlife is realized to allow processing at higher temperatures.

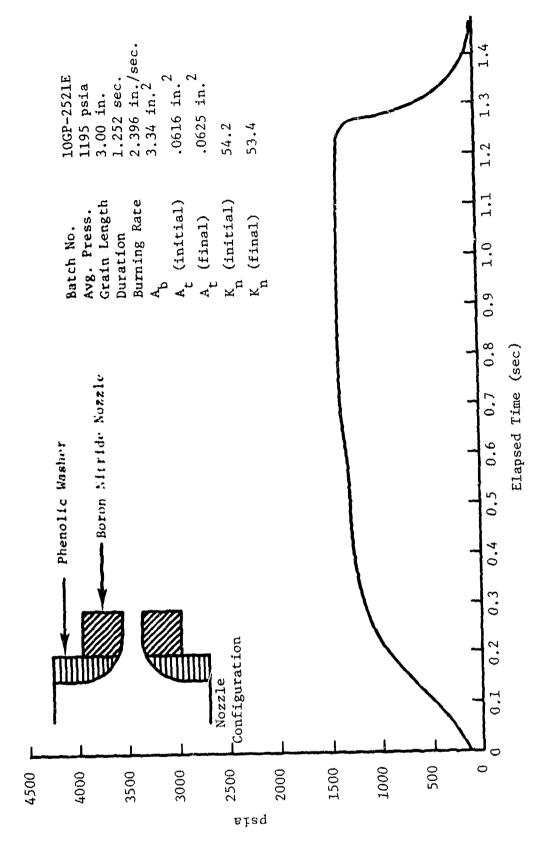
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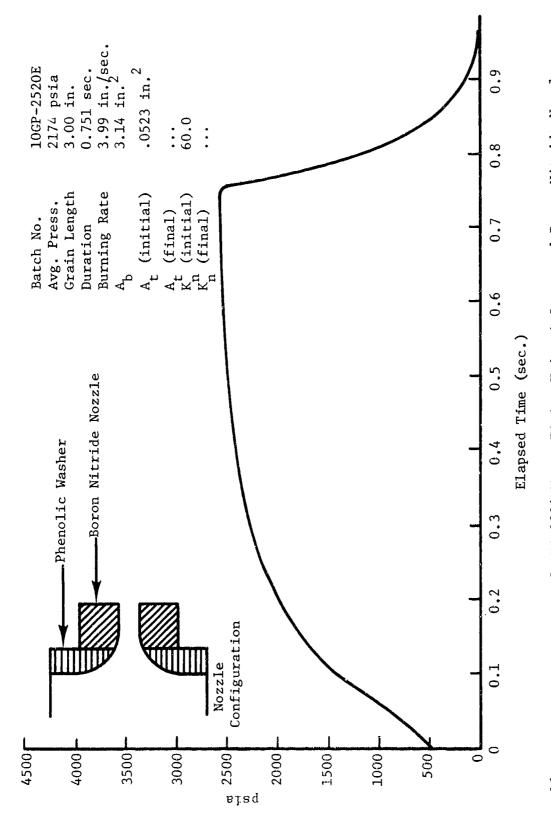
(U) FIG.14. Solid Strand and Motor Burning Rate Curves for ANB-3394 (A) and ANB-3395 (B) Propellant Formulations.

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(U) FIG. 15. Pressure-Time Trace of ANB.3394 Motor Firing Using A Contoured Boron Nitride Nozzle. A Non-Aluminized 0.25 in. First Fire Grain Was Cast Onto Main Grain to Prewarm Nozzle (P-T Trace Not Shown).

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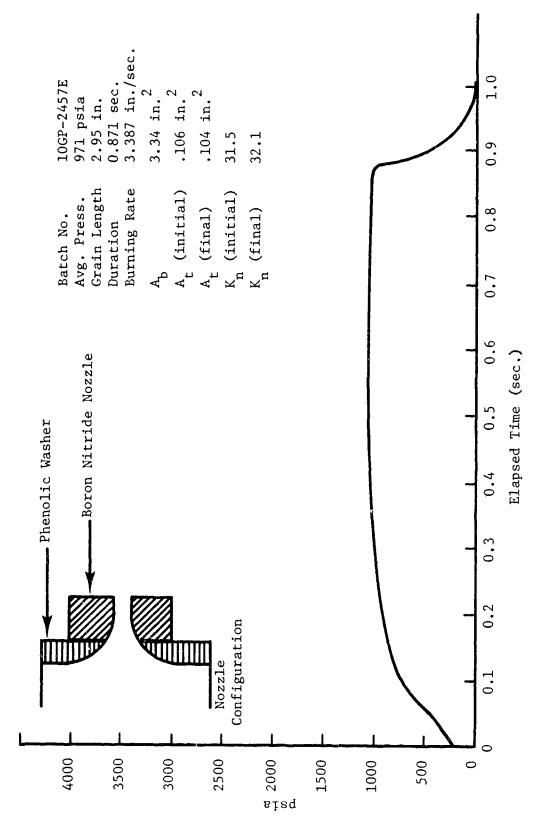


A Non-Aluminized 0.25 in. First Fire Grain Was Cast Onto Main Grain to Prewarm Nozzle (P-T Trace Not Shown). (U) FIG. 16. Pressure-Time Trace of ANB-3394 Motor Firing Using A Contoured Boron Nitride Nozzle.

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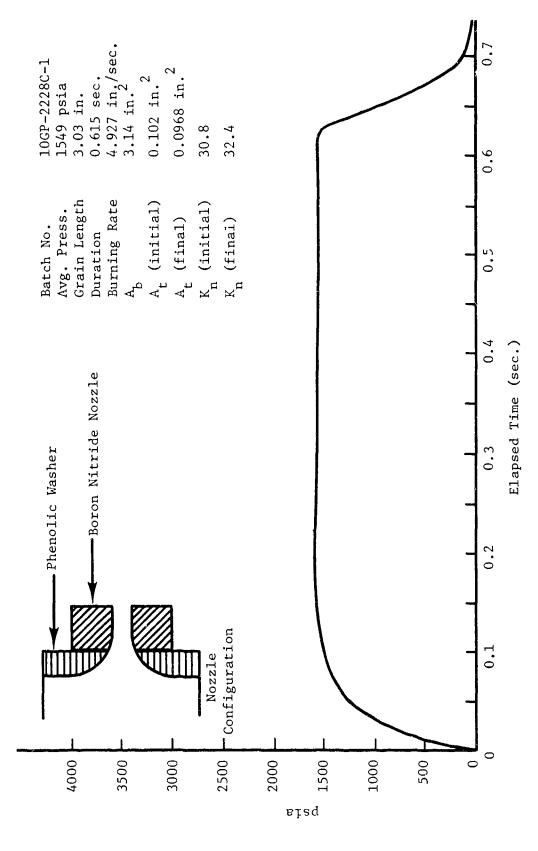
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A Non-Aluminized 0.25 in. First Fire Grain was Cast onto Main Grain to Prewarm Nozzle (P-T Trace not shown). (U) FIG. 17. Pressure-Time Trace of ANB-3395-1 Motor Firing Using a Contoured Boron Nitride Nozzle.

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18. Pressure-Time Trace of ANB-3395 Motor Firing Using A Contoured Boron Nitride Nozzle. FIG. (n)

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(U) TABLE 19. Summary of Initial and Aged Properties of ANB-3394

Processing and Cure Properties

	Initial	Aged 1 Month @ 135°F
Potlife, hrs. Shore "A" Hardness Density, g/cm ³	3.0 45 •••	 52 1.719

Mechanical Properties

	Initial			Aged 1	Month @	1.35°F		
!	160°F	77°F	0°F	-40°F	160°F	77°F	O°F	-40°F
σ _m , psi ε _m , % E _o , psi	43.3 18.5 275	72.5 20.8 453	131 18.3 956	270 14.8 2836	59.5 13.2 477	90.2 14.4 704	177 14.7 1490	329 13.0 3606

Ballistic Properties

	Initial	Aged 1 Month @ 135°F
R _B , in/sec		
500 psia 1000 psia 2000 psia	1.21 2.06 3.42	1.16 2.00 3.30
n (500-2000)	0.77	0.77

Liner/Propellant Bond Properties

	Initial	Aged 1 Month @ 135°F
DPT, psi	69.5 (P ₂ break)	92.4 (P ₂ break)

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(U) TABLE 20. Summary of Initial and Aged Properties of ANB-3395-1

Processing and Cure Properties

	Initial	Aged 1 Month @ 135°F
Potlife, hrs.	3.0	
Shore "A" Hardness	43	47
Density, gm	•••	1.729

Mechanical Properties

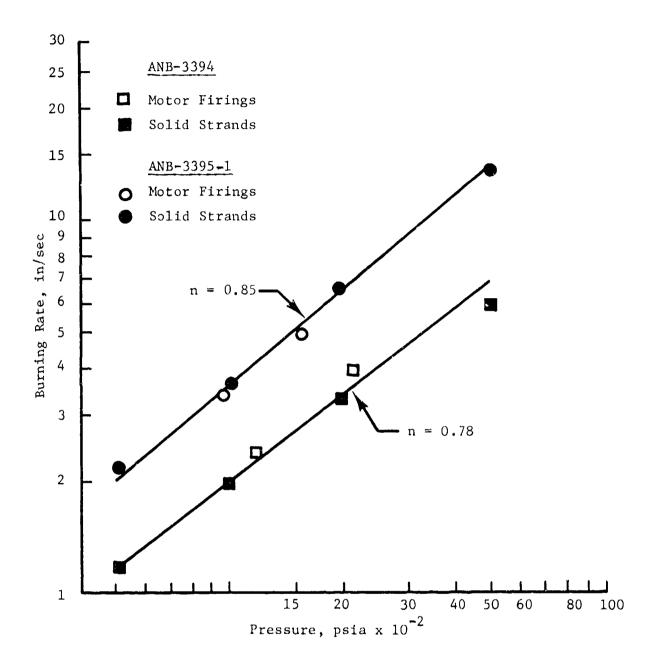
	Initial			Aged 1 Month @ 135°F				
	160°F	77°F	0°F	-40°F	160°F	77°F	O°F	-40°F
σ _m , psi ε _m , % Ε _O , psi	48.5 24.7 257	73.7 25.5 383	151 22.8 967	367 16.5 3650	62.9 23.7 337	88.1 22.8 528	192 19.8 1435	469 14.0 5123

Ballistic Properties

	Initial	Aged 1 Month @ 135°F
R _B , in/sec		
500 psia	2.18	2.13
1000 psia	3.55	3.49
2000 psia	6.80	6.81
n (500-1000)	0.71	0.72
n (1000-2000)	0.92	0.94

Liner/Propellant Bond Properties

	Initial	Aged 1 Month @ 135°F
DPT, psi	85.3 (P ₂ break)	106.7 (P ₂ break)



(U) FIG. 19. Burning Rate Curves from Solid Strand and Motor Rate Data for ANB-3394 and ANB-3395.

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Glossary of Terms and Abbreviations

Agerite White Antioxidant AO-2246 Antioxidant

AP Ammonium Perchlorate

BDB Aerojet proprietary coating agent

BRA-99 Aerojet proprietary combustion catalyst
BRA-101 Aerojet proprietary combustion catalyst

CTPB Carboxy terminated polybutadiene
DEO Hydroxy functional wetting agent

DOA Dioctyladipate

EDB Aerojet proprietary fuel component
ERL-4205 Bis(2-3-epoxycyclopentyl)ether

ERL-4221 3,4-Epoxycyclohexylmethyl-(3,4-epoxy) cyclohexane

carboxylate

FC-155 Aerojet proprietary fuel component

Freon-113 1,1,2 Trifluoro-1,2,2 Trifluoro-1,2,2 Trichloroethane

HC-434 Carboxy-terminated polybutadiene (Thiokol Chemical Co.)

HDI Hexamethylene diisocyanate

HTPB Hydroxy terminated polybutadiene

Hycat-6 A non-volatile liquid ferrocene derivative

IDP Isodecyl pelargonate plasticizer

IPDI Isophorone diisocyanate

Isonol Phosphorous containing polyol

MA Mikro-atomizer ground ammonium perchlorate

MSA Mine Safety Appliances Co., particle size measuring

apparatus, a liquid sedimentation technique

nBF n-Butylferrocene

P-33 Thermal carbon black

PAP Porous Ammonium Perchlorate

Plastinox 711 Antioxidant

R-45M Free radical initiated HTPB

Refrasil Silica Fiber

SS-AP Slow-speed mikro-pulverized ground ammonium

perchlorate

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Glossary of Terms and Abbreviations (Cont'd)

SURFAC OS Carboxy functional wetting agent

TEA Triethanol amine

TEHOS 2-Ethylhexylorthosilicate
Thixcin E Modified 1-hydroxy stearin

UFAP Ultra-fine ammonium perchlorate ($<5\mu$)

VEM Vibro-energy mill

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Propellant				:	i	
High Burn Rate						
Ferrocene				1	}	
Ultra Fine Ammonium Perchlorate		Ì				
Porous Ammonium Perchiorate		1	}			İ
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